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U.S. DEPARTMENT OF COMMERCE
National Bureau of Standards
National Measurement Laboratory
Center for Materials Science
Metallurgy Division
Washington, DC 20234

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Bureau of Medical Devices
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METALLURGICAL STUDIES OF INTERFACE BONDING ON IMPLANT ALLOYS

A. C. Fraker
A. W. Ruff
K. J. Bundy

with S. A. DeMontigny
P. Sung
A. C. Van Orden
K. M. Speck

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U.S. DEPARTMENT OF COMMERCE, Malcolm Baldrige, *Secretary*
NATIONAL BUREAU OF STANDARDS, Ernest Ambler, *Director*

Abstract

A literature review covering articles on stress analyses in total hip replacement prostheses and on the metal/bone cement interface is presented. The literature indicated the need for a test which utilized loading in torsion, and such a test was developed and is described. The test can be used to determine the influence of various parameters, including surface roughness and passivation and sterilization treatment on the strength of the metal/bone cement bond. Some preliminary tests were conducted and results are given. Future work using the mechanical test developed is discussed. Additional studies were conducted on the surface preparation of titanium, and data are presented to show that changes in initial electrochemical behavior and varying degrees of surface roughness occur depending on whether the metal receives a neutral, alkaline or acidic washing treatment.

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INTRODUCTION

This is a report of a one year effort which was planned and conducted to investigate the effects of surface treatments on surgical implant metals as related to interface problems between metals and bone cement in joint prosthesis fixations. It has been recognized for some time that prosthesis loosening is one of the most serious material problems confronting total or partial joint replacement today. While the cement sometimes is considered to be a grouting material, some adherence occurs between the bone and cement, and between the cement and the metal. The importance of this adherence is verified by the fact that the x-ray radiographic appearance of a line separating either bond is a prelude to failure of the device. This failure can be due to fatigue and fracture, or overload fracture which can lead to pain and malfunction of the device.

Recently, at Northwestern University, a study was made of several factors affecting surgical implant metal/bone cement adhesion. Those data indicated that surface treatments on metals such as electropolishing and passivating resulted in the strongest adhesive bond when joined to the cement at the onset of the dough stage (after 3 minutes). It is clear from this work that much useful information can be obtained from studying these interfaces. To date, very little data exists on the nature of the metal/cement interface and the bond strength. Measurements of the surface roughness on the metal and the surface film chemical composition should be made and correlated with mechanical strength of a cement bond to the given metal. These measurements can contribute data for use in standards and tests methods which can provide improved quality

assurance against device loosening and more adequately assure safety and long term usefulness of the device.

A review of literature and other documented information on surface treatments and test methods for bond strength has been completed and is summarized in Part II by Dr. Kirk Bundy, of the Johns Hopkins University, who worked at NBS on this project. Over 200 published articles pertaining to bone cement and cemented prostheses were collected and reviewed. In addition, hundreds of other references which related to other considerations in prosthesis installation were reviewed. These articles are now part of a reference file containing over 1000 articles pertaining to skeletal implants.

The literature review indicated that several factors affect the strength of the bone cement/metal interface bond including the type of bone cement and the surface conditions of the material. It is clear that several fracture and fatigue test results on the bone cements are not in agreement. One study indicates superior fracture toughness and fatigue behavior for one type of bone cement while the other study shows that a different bone cement is superior in these qualities. A third study discussed some differences in the cements but did not indicate differences in mechanical strengths.

Alternative methods of prosthesis fixation are being considered and developed. However, there are many patients who always require a cement fixation of the prosthesis. Metal porous coatings on metal prostheses are being studied for use in cementless prostheses. Porous metal, porous polymer, ceramic, and bone cement coatings could be applied to a prosthesis in selected areas where the forces on the bone cement/metal interface are the greatest such as the upper side of the proximal end of the femural component.

Most manufacturers follow the procedures given in the ASTM F4, F-86, document entitled "Standard Recommended Practice for Surface Preparation and Marking of Metallic Implants." Stems of hip prostheses often are given a satin finish by dry blasting with Al_2O_3 and/or glass beads. The articulating areas are highly polished. Prostheses are cleaned as described in F-86. There are steps in the procedure where differences can occur such as washing after different treatments. The final steps are passivation and sterilization. NBS studies of the surface preparation and corrosion behavior of titanium and titanium alloys were presented in a paper at the ASTM Symposium on Titanium for Surgical Implants, May, 1981 and are presented in Part III of this report.

The test methods of ASTM D-14 on adhesives were reviewed (see Part II) and found not to be appropriate for testing the bone cement/metal interface. An appropriate test method was developed to the point of application for measuring bond strength of the cement/metal interface to test various surface related parameters. The test is described in detail in Part II. This test will be used in the continuation of the metal/bone cement interface studies

Part II

INVESTIGATION OF STRESS ANALYSES AND THE METAL/BONE CEMENT INTERFACE IN TOTAL HIP REPLACEMENT PROSTHESES

Kirk J. Bundy

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CHAPTER I

INTRODUCTION

This report describes activities pursued in the Metallurgy Division of the National Bureau of Standards to study the influence of the metal/bone cement interface on the performance of prostheses. The rationale for this work is described below.

In recent years the field of surgical implants has become increasingly important for a number of reasons. One reason is that the average age of the population is increasing and thus greater numbers of individuals are in need of implants. Another is that devices are being implanted in younger individuals more often now than previously was the case. Therefore, these devices will serve within the body's environment for longer periods of time. A case in point involving a particularly demanding application is the artificial hip.

As a consequence of these and other trends, there is increasing interest in the factors which govern the success and failure of orthopedic implants. This subject is a complicated one and involves both physiological and engineering effects. In the latter category, corrosion of the implant metals and the stress distribution in the implant--bone cement--bone region have been related to implant failures. The interfacial regions are particularly prone to loosening and failure. Another aspect of the matter is that alteration of the stresses in bone due to the presence of implants can occur which may lead to adverse physiological consequences.

In order to gain further insight into these problems, the research described in this report, mainly a literature search, was undertaken. In Chapter II the effects on bone of alterations of the natural

physiological stress distribution caused by the presence of implants and other means is described. Chapter III is concerned with a review of stress analyses of total hip replacements. Both experimental and theoretical studies are examined. Particular attention is paid to analyses of the stresses at the bone cement/metal interface.

Chapter IV reviews clinical reports of failed bone cement/metal interfaces and compares them with the results of stress analyses. The lack of correspondence between the two suggests that torsional forces which act at the interface could be important in its failure.

Chapter V reviews methodology for testing interfacial bond strengths. ASTM standards are reviewed and the literature pertaining to mechanical testing of bone cements and cement/metal interfaces is discussed. Finally, experimental results of preliminary pilot studies on measuring torsional strengths of bone cement/metal interfaces are described. Conclusions and recommendations are given in Chapter VI.

Chapter II

EFFECT OF ALTERED STRESS DISTRIBUTION ON BONE

Background Information

One of the main reasons for interest in stress analyses of total hip replacements is, in addition to insight into failure of the components themselves, to determine the stresses present in the surrounding bone. Maintenance of an adequate stress level in skeletal structures is probably the major factor in maintaining the normal balance between bone formation and resorption processes (Tonino et al., 1976). This relationship was first pointed out in 1870 (Wolff, 1870) and has since become known as Wolff's law which can be stated (Pugh et al., 1973):

> ... changes in stress of the bone induce changes in its structure appropriate to support the changed load.

Unfortunately Wolff's law is a qualitative principle and not a quantitative relationship which can be used to predict a reduction of bone mass or density which results from a given reduction in bone stress level. If Wolff's law was a quantitative relationship, much additional insight into bone's response to stress could be obtained from stress analyses such as those described in Chapter III.

Various mechanisms to explain Wolff's law have been proposed. Bone is considered by some researchers to be a piezoelectric material (Fukada and Yasuda, 1957), (Shamos and Lavine, 1964). It has also been suggested that physical movement of intercellular fluid over the charged surfaces of cells, which is caused by mechanical stress, induces streaming potentials (Dumbleton and Black, 1975). It has been proposed that the electric fields caused by applied loads (or absent in the absence of load) act as "signals" which trigger osteoblastic

and osteoclastic activity and regulate the rates of bone production and resorption. Bone in its response to stress has been compared to a system controlled by a feedback loop (Kummer, 1972). It has been demonstrated that even in the absence of stress, applied fields of the appropriate voltages, current levels, and frequencies stimulate bone growth (Dumbleton and Black, 1975).

In cancellous bone which was caused to remodel by experimental loading, Pugh, Rose, and Radin (1974) observed significant fracture of trabeculae on a microscopic scale. The healing of these fractures produces large amounts of callus which serves to stiffen the bone. They propose that this mechanism could underlie Wolff's law and could be responsible for cancellous bone remodeling in response to changes in stress patterns.

Whatever the mechanism behind Wolff's law, it is clear that absence or reduction in stresses can lead to adverse consequences to skeletal tissue. This seems to be true no matter how the stress reduction comes about: through weightlessness (as with astronauts), limb paralysis (as in poliomyelitus), bed rest, or stress redistribution due to implants.

The time scale required for a stress level reduction to affect bone can be quite short. The bones of astronauts in weightless environments undergo demineralization (Kummer, 1972). The effects are measurable after fourteen days in space (Kazarian and Von Gierke, 1969). On the other hand, as discussed in the next section, a longer term consequence of reduction in stress, such as that which occurs in the bone around implants, is resorption.

Implant Induced Response of Bone to Changes in Stress

When a load-bearing metal component is introduced into bone tissue, a reduction of the load transmitted through the adjacent bone will occur. To a certain degree this is desirable. For example, fracture fixation plates function by partially taking the load off the fractured pieces of a bone and rigidly holding them in place so that healing can occur.

This reduction in load comes about since most implant designs will produce a situation in which the displacements of the surrounding bone due to externally applied forces will be constrained to be either equal to those in the stiffer implant or at least reduced from those present when an intact bone is loaded. The result is a decrease in strains in the bone and thus a decline in stress levels also. This effect is often called "stress shielding" or "stress protection".

Due to the action of Wolff's law stress shielding can have a number of undesirable consequences for bone tissue as discussed below. In fracture fixation plates, the need for high stiffness to insure stability for healing and the need for low stiffness thereafter to avoid the harmful results of stress shielding pose an unresolved dilemma in orthopedic surgery.

For an implant of the same material and shape, Woo et al. (1977) have used finite element calculations to demonstrate that the stresses in bone diminish as the cross-section increases. Also, it has been experimentally shown that for implants of the same size and shape, the stresses diminish as the modulus of elasticity of the material increases. Woo et al. (1976) conducted 9 to 12 month studies of implants made from a graphite fiber methyl methacrylate composite

resin and from a Co-Cr alloy. They found that stress shielding did not affect the mechanical properties (ultimate bending strength, energy absorption capacity, and flexural modulus of elasticity) of bone as a tissue material but did adversely effect the bone's ability to bear load due to a change in its structural properties, i.e., thinning of the cortex. Significantly less severe effects were noted for the more flexible nonmetallic material.

In a somewhat similar study Tonino et al. (1976) have demonstrated that bone mass per unit length decreases due to both trabeculation of previously compact areas of bone at the endosteal surface and to decreased mineralization of newly formed bone in this area. These results were obtained after 10 to 18 weeks of implantation of stainless steel or polytrifluoromonochloroethylene in dog femora. Corresponding reductions were observed in effective ultimate bending strength and effective Young's modulus (as determined from tests on whole bones). These effects were more pronounced with the steel. Slätis et al. (1980) have observed cortical thinning and development of porosity near dynamic compression plates and have measured corresponding reductions in torsional loads required to produce fracture.

Observations of disuse osteoporosis beneath plates appear often in the literature and have also been noted by Dodge and Cady (1972), Matter et al. (1974), Olerud and Karlstrom (1972), Richon et al. (1967) and others. A number of investigators (Cochran, 1969), (Köbler and Wiechell, 1971), (Uhthoff and Dubuc, 1971), (Diehl and Mittelmeier, 1974) have suggested that the osteoporosis produced by stress shielding leads to increased likelihood of fatigue fracture after removal of the plate. A well recognized complication of the use of plates for internal

fixation is a refracture or a fracture through the porotic portion (Chrisman and Snook, 1968), (Dencker, 1964), (Frankel and Burstein, 1968), (Solheim, 1972).

Since there is a wide disparity between the Young's moduli of the commonly used implant metals and bone (see Table II-1) and larger, thicker, more rigid plates are growing in popularity (Moyen et al. 1978), (Müller et al. 1965) it can be expected that reports of harmful effects due to stress shielding by fracture fixation plates will continue to occur in the future. The development of composite materials with elastic properties more closely matched to bone and/or development of higher strength alloys which can be used in thinner sections could represent a long term solution to this problem.

Problems with regard to stress protection or shielding are not confined to fracture fixation plates. One way in which total hip replacements fail is by fracture of the femoral stem. Lack of bony support in the area of the calcar femorale seems to occur often in cases of stem fracture--always according to Galante (1980) although Collis (1977) found other causes related to heavy patient weight, inadequate valgus positioning, and metallurgical inadequacies.

Whether or not the ultimate result is stem fracture, resorption in the medial area of the femoral neck is common in total hip replacements. Observed frequencies have been 16.8 percent (Beckenbaugh and Ilstrup, 1978) and 21 percent (Bocco et al., 1977). Inadequate transfer of load from the implant to the calcar due to lack or inadequacy of physical contact between them has been suggested by several investigators as a biomechanical cause for the resorption (Galante, 1980), (Oh and Harris, 1978), (Markolf et al., 1980). These investigators have

also mentioned other possible causes however: a relative ischemia (i.e., a subnormal amount of blood) of the area or tissue reaction to bone cement wear particles. Another nonbiomechanical cause for resorption has been proposed by Smethurst and Waterhouse (1978). In a study of failures of metal-on-metal total hip replacements, bone loss was attributed to corrosion of the components which resulted in release of Cr and Co ions.

TABLE II-1

YOUNG'S MODULUS OF SURGICAL IMPLANT MATERIALS AND BONE

Material	Young's Modulus (10^3 MPa)
Bone	20
316 L Stainless steel	200
Cast Co-Cr alloy	200
Wrought Co-Cr alloy	230
MP35N	230
Pure Ti	100
Ti-6Al-4V	100
PMMA bone cement	2

after Swanson and Freeman (1977)

Chapter III

A REVIEW OF STRESS ANALYSES AND MEASUREMENTS OF TOTAL HIP IMPLANTS

Many studies have been conducted to obtain stress analyses of total hip replacements for purposes of design improvements or development of understanding of the biomechanical basis of clinical failures. This work is reviewed in this chapter.

To perform a theoretical stress analysis, the following information must in general be known: (1) the geometry of the structure of interest, (2) its relevant mechanical properties, in particular, its elastic properties, (3) the loads applied at its surface or the surface displacements, and (4) appropriate boundary conditions.

An exact stress analysis of a total hip replacement procedure which provides information about the stress state at all points in the metal prosthesis, bone cement, bone itself, and at the interfaces between them is an exceedingly difficult and perhaps almost intractable task because of difficulties associated with all four areas mentioned above.

The complexity of this problem can be appreciated by examining Figure III-1. The geometry which is involved is irregular and may require a three dimensional treatment of the problem. There are three different materials whose elastic properties are important: bone, bone cement, and the alloy of the femoral component. The properties of the cement can vary considerably depending upon its handling and various environmental factors. In the case of bone (at least) the situation is further complicated by the spatially nonuniform and anisotropic nature of the tissue and the presence of both cortical and cancellous

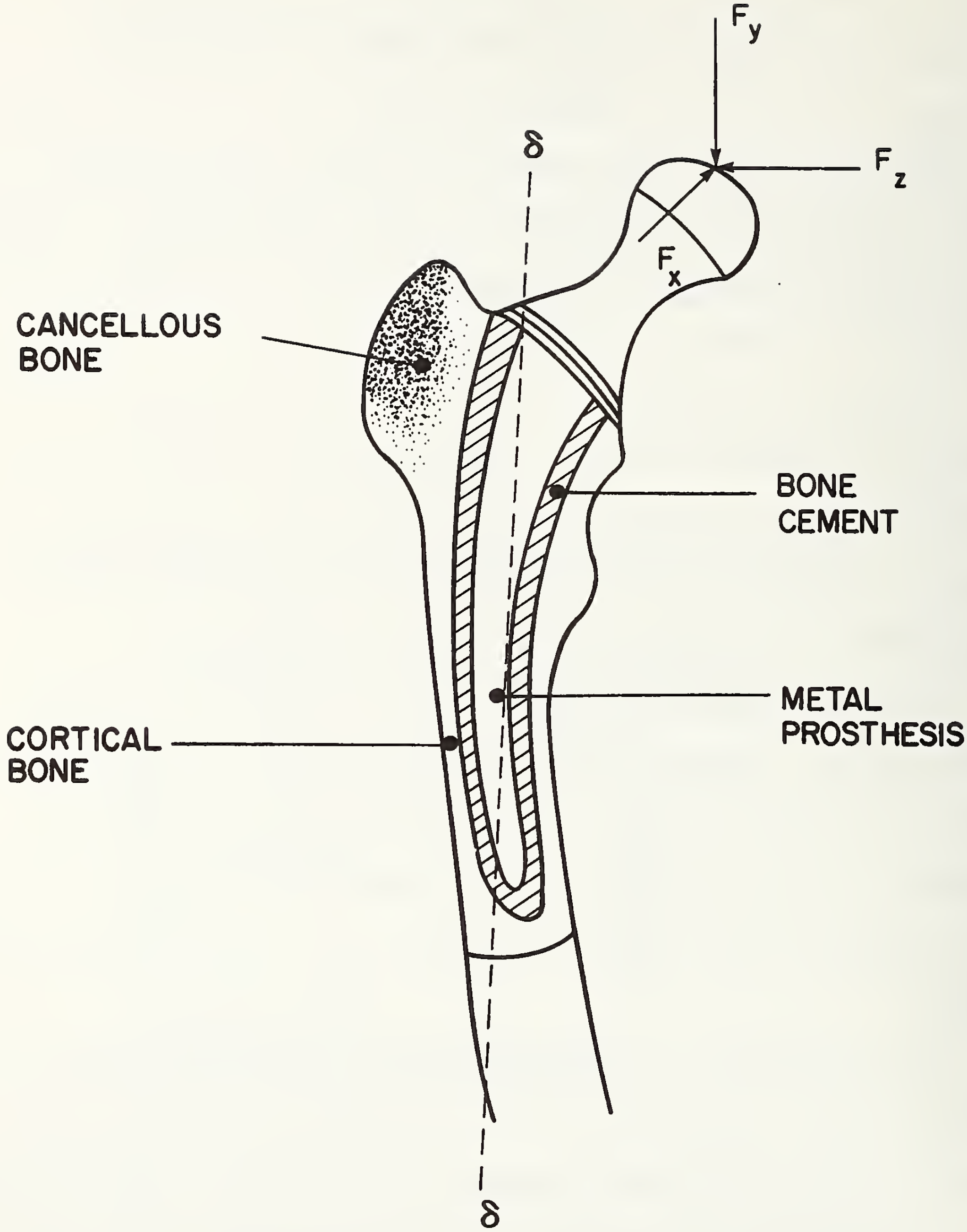

FIG. III-1 Total Hip Replacement Prosthesis and Applied Joint Force Components

bone. The loads in the hip joint which are applied to the prosthesis have components in all three coordinate directions which vary with time, and a consensus does not appear in the literature as to what force state should be used in calculations. As will be discussed later in the report, the boundary conditions which should be used to model the bone/bone cement and metal/bone cement interfaces are also uncertain.

It is thus not surprising that a variety of methods have been used for hip implant stress analysis and that no agreement has yet emerged as to which is the most valid for this purpose. In what follows these methods are first very briefly described. Then the results of representative studies which use these techniques are discussed. Although these methods are broadly applicable to a great number of problems dealing with total hip replacements, the topic of most interest to this report is the stresses which exist at the metal/bone cement interface.

Methods Used to Investigate Stresses in Hip Implants

The basic principles behind and, where applicable, the limitations of the various methods used to determine stresses in hip implants are given below:

Theoretical Methods

1. Finite Element Analysis: The differential equations which govern the load-displacement behavior are numerically solved at a finite number of points (called nodal points) in an irregularly shaped object and the behavior at the other points in the object are interpolated from this information. Stresses and strains are derived from this data. Anisotropic elastic properties and spatial change in elastic properties can be straightforwardly included in this type of analysis.

2. Beam Theory: In beam theory as applied to problems of concern to surgical implants (Bartel, 1977), (Barberi et al., 1978), simplifying assumptions are made with respect to deformation (i.e., that plane sections remain plane) and the materials used (which are assumed to be homogeneous, isotropic, and linearly elastic) so that closed form solutions for stresses and bending moments in bone, bone cement, and/or the metal stem can be determined along the length of the prosthesis. Variable geometry and elastic properties along the length are taken into account with the beam theory approach. Joint loads which produce torsional stresses have not yet been examined with this method.

Experimental Methods

1. Strain Gauge Techniques: Strain gauges which consist of several loops of foil or wires whose electrical resistance is known as a function of deformation are bonded to the object to be measured at points of interest. Loads are applied. By monitoring the change in electrical resistance of the strain gauges with a Wheatstone bridge circuit, the normal strains at the surface of the object are determined. Shear strains can be determined indirectly if the gauges are placed in a "rosette" pattern (Dieter, 1976). Stresses can be calculated for elastic behavior assuming that the elastic properties are known.

2. Photoelastic Methods: A model with the geometry of the object of interest is constructed from a transparent optically birefrigent plastic, (i.e., a material with a refractive index which depends upon the plane of polarization of the light which is incident upon it). The loads of interest are applied and an optical

interference pattern is developed when plane polarized monochromatic light is passed through the object and viewed through an analyzer (Kolsky, 1963). The stress distribution is inferred from this fringe pattern.

Problems Examined by Stress Analysis of Total Hip Replacements

Before considering in detail results which have been obtained with the various stress analysis techniques, a brief overview of the different classes of problems which have been investigated is given below.

A number of studies have been concerned with the stresses in the materials of the total hip implant as they are influenced by various design parameters e.g., stem length, elastic modulus of the femoral component and the bone cement, stem shape, presence or absence of a collar resting upon the medial shoulder of the bone, and presence or absence of the bone cement layer. Among the studies which fall into this category are those of Svensson et al. (1977), Cook et al. (1980), Scholten et al. (1978), Yettram and Wright (1980), Mizrahi et al. (1979), and Barberi et al. (1978). Most studies have mainly been concerned with determination of the stresses in the metallic component and/or the surrounding bone. Some studies, however, have been concerned (at least in part) with the stress distribution in the acrylic bone cement (Svensson et al., 1977), (Yettram and Wright, 1979), and (Kennedy et al., 1979).

Another important class of problems which has been investigated is that which involves attempts to provide biomechanical explanations for different types of failures of total hip implants which have been observed clinically. Topics which have been examined include the

desirability of valgus as opposed to varus orientation for the stem, stress shielding in the calcar region leading to bone resorption and stem loosening, increases in distal stem stresses due to inadequate proximal medial support, fracture of the acrylic or interface due to high stresses resulting in stem loosening, and the importance of the various force components in causing failure. Some studies in this category are those by Hampton et al. (1980), Dobbs and Chaplin (1981), Markolf and Amstutz (1976), Jacob and Huggler (1978), and Jacob and Huggler (1980).

Finally, it should be mentioned that some studies have been concerned with comparing a theoretical approach (the finite element method) with experimental results (Svensson et al., 1980), (Rohlmann et al., 1980). Some work has also been done with regard to stress analysis of intact bones subjected to joint loads (Toridis, 1969), (Rybicki et al., 1972), (Valliapan et al., 1977) and (Piotrowski, 1975).

<u>Results of Experimental Measurements of Total Hip Replacements</u>

Some measurements have been made of the gross response of total hip replacements to mechanical forces. McCarthy and Wells (1977) loaded prostheses (which had been implanted in dogs) in the axial direction and measured the maximum load. They divided this number by the implant surface area to find the "shear strength" of the bone/bone cement interface to be 1.49 ± 1.27 MPa. Markolf et al. (1980) measured displacements of the collar and tip of femoral components implanted in cadavers due to a 2000 Newton femoral head force.

Studies which have involved more refined measurements have been concerned with determining the stress distribution in the components of the total hip replacement. Kennedy et al. (1979) used photoelastic

techniques to study the stress distribution in bone cement, a factor which they considered important in regard to loosening of the femoral component in its bed of cement and for understanding nonuniform load transfer to the femur which could result in resorption. The models were constructed from actual hip prostheses, bone material or aluminum to simulate the femur, and an epoxy with elastic properties similar to bone cement. They found that a porous coated stem was superior to a smooth stem with respect to promoting a more uniform stress distribution and that porous coatings prevented loosening of the stem in the cement.

Mizrahi et al. (1979) used strain gauges to measure the stresses at the surface of Straight Narrow Stem and Standard Charnley prostheses. They compared their results to calculations using curved beam theory and found good agreement. The main factor they investigated was the stresses which result from applied forces which correspond to different anatomical positions of flexion-extension and abduction-adduction. They found that both normal stresses and shear stresses were maximum at a position corresponding to 20° flexion and 10° abduction and are comparable to the fatigue limit of the material. The shearing stresses which correspond to twisting or torsion of the prosthesis in the cement are highest near the proximal end and increase with the degree of flexion and abduction.

Jacob and Huggler have written two papers (1978) and (1980) in which strain gauge measurements were made on hip devices implanted in epoxy models to simulate bone. The aim of this work was to clarify the causes of stem loosening in the proximal end of the femur. They found out that a prosthetic hip replacement could greatly diminish the stresses in cortical bone at the proximal end of the femur such that

in some cases the stress level is reduced to 40 percent of that present in the physiological state. Also, it was shown that a stem which had loosened in the bone was a very adverse situation which creates high bending stresses in the prosthesis and could cause stem breakage. Although the presence of cancellous bone between the cortical bone and bone cement could exercise a favorable load distribution function, the possiblity exists that the stresses can be sufficiently high to cause fracture of trabeculae. Direct bearing of a stem neck collar on the underlying cortical bone of the calcar did not produce significant alteration of stresses within either the stem or the bone.

Two studies, (Dobbs and Chaplin, 1981) and (Markolf and Amstutz, 1976), are directly relevant to the main subject of this report. Markolf and Amstutz used strain gauges to measure stresses at the surface of metal prostheses potted into 6 cm diameter cylindrical molds of PMMA. Both cantilever loading, vertical loading, and loading in which the downward force vector was inclined form the vertical at angles of -3, +17, and +37° were studied. They found that varus positioning of the stem should be avoided at surgery because it produced marked increases in stem stress. Varus orientation is when the ball of the prosthesis at the proximal end is tipped medially and the distal end of the stem is tipped laterally. They also showed that lack of adequate support in the proximal medial region, due to acrylic fracture or poor bone quality creates dangerous stress levels in the stem. Finally, their finding which is most directly pertinent to this study was that a prosthesis which is loose in the bone cement causes an extremely severe rise in stem stress level. At the distal end of the stem, the stress levels are more than one order of magnitude greater for stems in which only the distal 1/3 of the stem is fixed in acrylic

as compared to a stem in which the total length is rigidly fixed. They concluded from this that it is very important during hip surgery to avoid movement of the prosthesis within the acrylic during the time that the bone cement is setting. It presumably could also be concluded from this data that materials treatment techniques which would promote higher strength bonds between the metal and bone cement would be desirable.

Dobbs and Chaplin (1981) tested femoral components embedded in acrylic which was contained in a thin walled cylindrical fixture. Strain gauges were used to measure the strains in the acrylic, stem, and fixture. It was demonstrated that the technique gave results similar to those observed for prostheses embedded in actual femurs. They studied loads acting in the medial-lateral plane at 0°, 10°, and 20° from the vertical. Here also, high stresses were observed for varus positioning. Most importantly they observed high stresses in the acrylic which they felt were high enough to cause acrylic fatigue fracture and interface failure. They recommend that future designs of prostheses should attempt to reduce bone cement stresses and increase interfacial strength.

Results of Beam Theory Applied to Total Hip Replacements

Beam theory seems to have been more applied to calculating stress distributions in the natural femur itself than to analysis of total hip replacements. Although tangential to the emphasis of this report a few of these studies will be mentioned briefly. The first of these was the work of Koch (1917) who used beam theory to verify Wolff's ideas (1892) of bone architecture. Toridis (1969) introduced the mathematical refinement of using the theory of curved beams to analyze these problems. Finally, Rybicki, Simonen, and Weis (1972) compared

beam theory to the finite element method. Their findings indicated that beam theory was appropriate for the shaft of the femur, but that the more complicated continuum elasticity theory was required in the areas near muscle attachments and geometrical irregularity such as the femur head and greater trochanter. This would be expected due to Saint Venant's principle (Papov, 1976).

Two investigations have used beam theory for hip implant problems. Bartel (1977) calculated stresses in bone, bone cement, and the prosthesis as they are affected by diameter of the prosthesis stem and medullary cavity. Calculations of stresses along the length for both bone and the prosthesis are presented. Barberi et al. (1978) used beam theory applied to the actual geometry based upon Legendre polynomials, measured an interfacial stiffness experimentally, and computed the stress state which minimized the strain energy. Torsional loads were neglected. They computed the distributed compressive force and flexural moment in the bone and the maximum flexural stress in the stem for nine combinations of stem stiffness and length. They conclude that longer stems do not necessarily lower all the stresses in the cement and that, due to the nature of the flexible support of the femur, stiffer stems do not have lower stresses.

Apparently beam theory studies, while of some use in understanding stresses in implants, have been superceded by the more technologically sophisticated methods of strain gauge instrumentation and finite element method computer analysis. It should be pointed out, however, that in at least one investigation, beam theory calculations corresponded more closely to the results of rigorous three dimensional finite element analysis than did a simplified two dimensional finite element analysis (Valliappan et al., 1977).

Approaches Used for Finite Element Analysis of Total Hip Replacements

A number of different investigators have applied the finite element method to various problems of interest with regard to total hip replacements. With some exceptions, e.g. Yettram and Wright (1979) and Svensson, Valliappan, and Wood (1977), they have not been concerned with the stresses at the interface.

Table III-1 summarizes the modelling approaches used by some of these investigations. Specific details of the results are described in the next sections. Some aspects of these studies are quite similar, but there are also significant differences. For example there is no consensus as to the best way to model the boundary condition between the femoral stem and the bone cement. Yettram and Wright (1979) use a displacement continuity condition. This is equivalent to perfect adhesion at the interface with no slip between the two components. Svensson et al. (1977) employ a no-slip condition on the medial and lateral surfaces and a no-adhesion condition on the anterior and posterior surfaces. Also, in this regard it should be pointed out that another study of the biomechanical behavior of the hip implant has considered the boundary condition to be that of a perfect grouting material, i.e., frictional forces at the interface (Berme and Paul, 1979). Some investigators have used two dimensional finite element models and others employ three dimensional models. In principle, the three dimensional approach is more mathematically rigorous (and much more complex to use) and should give a better approximation to reality. There also does not seem to be consensus as to what joint loads should be used for finite element analysis. A vertical load or the forces present in a one-legged stance seem to be the most common choices.

TABLE III-1

VARIOUS APPROACHES EMPLOYED FOR FINITE ELEMENT ANALYSIS OF TOTAL HIP REPLACEMENTS

A. Type of Model

Investigators	Model
Yettram and Wright (1980)	2 dimensional, plane stress, 751 isoparametric quadrilateral elements, 834 nodes, orthotropic elastic properties used for bone
Yettram and Wright (1979)	Same as above
Cook, Klawitter, and Weinstein (1980)	2 dimensional, plane stress, 500 constant strain triangular and quadrilateral elements, 524 nodes, isotropic elastic properties used for bone
Scholten, Röhrle, and Sollbach (1978)	3 dimensional, tetrahedron volume elements with square displacement function at the edges, isotropic elastic properties for bone with spatial nonuniformity in properties taken into account
Hampton, Andriacchi, and Galante (1980)	3 dimensional, 31 isoparametric hexahedron elements, 200 nodes, isotropic elastic properties for bone
Svensson, Valliappan, and Wood (1977)	2 dimensional, 118 quadrilateral elements, isotropic elastic properties for bone
Svensson, Valliappan, and McMahon (1980)	3 dimensional, 167 isoparametric elements with 20 nodes each

B. Boundary Conditions

Investigators	Interface Assumptions
Yettram and Wright (1980)	not explicitly given
Yettram and Wright (1979)	displacement continuity, i.e. no slip, at the metal/bone cement and the bone/bone cement interfaces
Cook, Klawitter, and Weinstein (1980)	not explicitly given; elements between the metal prosthesis and bone itself were given different values of elastic properties to simulate different fixation mechanisms such as bone cement and porous coatings

Table III-1 B. (continued)

Scholten, Röhrle, and Sollbach (1978)	this was a variable investigated in the study; both displacement continuity and tangential displacement of prosthesis relative to bone were examined
Hampton, Andriacchi, and Galante (1980	displacement continuity at both interfaces
Svensson, Valliappan, and Wood (1977)	displacement continuity at the medial and lateral bone cement/metal interfaces; non-adhesive, i.e. slip, conditions for the anterior and posterior interfaces
Svennson, Valliappan, and McMahon (1980)	Goodman joint elements which correspond to slip and no normal stress transmission through the element for tensile contact stresses

C. <u>Type of Loading</u>

Investigators	Joint Forces
Yettram and Wright (1980)	10^3 lb vertical load
Yettram and Wright (1979)	same as above
Cook, Klawitter, and Weinstein (1980)	1250 N acting at 20.5° to vertical at the femur head, 778 N acting upward at 22° to vertical at greater trochanter (abductor muscle traction); i.e. the one-legged stance
Scholten, Röhrle, and Sollbach (1978)	the one legged stance of Pauwels (1965); femur axis inclined 9° to vertical, joint reaction force 175 kgf at 16° to vertical, 130 kgf abductor muscle traction at 21° to vertical
Hampton, Andriacchi, and Galante (1980)	stresses normalized to a 1 N load in each of the 3 coordinate directions (vertical, anterior-posterior, and medial-lateral) were calculated
Svensson, Valliappan, and Wood (1977)	the one legged stance of McLeish and Charnley (1970); 1620 N downward at 24° to vertical as the joint reaction force, abductor traction of 1062 N upward at 29° to vertical, a second muscle traction of 265 N vertically downward at the trochanter
Svensson, Valliappan, and McMahon (1980)	3 kN downward vertical load acting at 7° to axis of prosthesis

Muscle tractions are sometimes neglected, and only static situations are considered.

Results of Finite Element Method Studies of Total Hip Implants

A number of studies have been conducted with the finite element method which focus on various aspects of implant design. Cook et al. (1980) studied the factors which promote a satisfactory stress distribution in the calcar region. Noting the current trend toward larger stem sections to prevent fatigue failures of the stem, they point out the possibility of stress shielding and therefore bone resorption at the calcar region. The resorption can cause implant loosening proximally and therefore higher stresses distally which still could lead to fatigue failure. As would be expected for a material of lower stiffness which would be less prone to stress shielding, their results indicated that hip implants made of carbon would produce a higher, more physiologically normal stress pattern in the calcar region. It also was shown that shortening the length of the stem did produce higher average stresses but the loading pattern was not physiological.

Scholten et al. (1978) studied stresses in the surrounding bone for eight different fixation combinations involving support or non-support at the prosthesis shoulder area (i.e., with or without a collar); direct stem to bone contact or stem to bone contact through an intervening layer of bone cement; and sliding or displacement continuity boundary conditions. Most of the calculations involved stress in the bone although some study of the stresses in the prosthesis itself was carried out. A prosthesis of conventional design was compared to a Ritter-Grunert prosthesis which has a tension bar through the trochanteric region which is connected to a fixation plate. They

found high compressive radial stresses, high tensile tangential stresses, and axial compressive stresses in the bone on the medial side for sliding boundary conditions (as would be expected from a prosthesis acting as a wedge). They also calculated the stress distributions in the natural bone and found that the Ritter-Grunert prosthesis produced a stress distribution much closer to the physiological state than did the conventional prosthesis.

Additional studies have been concerned with the stresses in the metal stem component itself. Hampton et al. (1980) used a three dimensional model and determined the longitudinal stress per unit load in the stem of hip implants for loads directed, respectively, along the x, y, and z coordinate axes (see Figure III-1). The stresses are highest in the region of the implant about 1/3 to 1/2 of the way along the implant from the distal end. Their conclusion was that the vertical load was responsible for most clinical failures. However, the lateral and posterior loads were significant enough to possibly endanger survival of the device. The posterior load (which can be high in activities such as stair climbing, rising from a chair, or in certain phases of the gait cycle, see Figure IV-1) when combined with the vertical load produces significant stress in the anteriolateral corner of the stem, a site at which cracks often originate in failed devices. This failure site has also been described by Harris and Tarr (1979).

The hypothesis advanced in the next chapter is that the posterior force is important to understand the failure of the metal/bone cement interface. Unfortunately no information on this subject is available from Hampton's study because it was optimized to determine stresses in the metal rather than in the acrylic.

Yettram and Wright (1980) used a two dimensional finite element model (which was found suitable for parametric studies) to calculate the tensile stresses along the lateral surface of the stem of the femoral component of the total hip replacement. The variables examined were stem taper, stiffness of the cement, stiffness of the prosthesis, and the presence or absence of a shoulder on the prosthesis. It was found that cement stiffness and implant shape had a minor effect. The Young's modulus of the stem material was found to be the most important factor. Decreasing stress levels were observed in less stiff implants (as would be expected from a simple consideration of stress shielding effects).

Yettram and Wright (1979) in another study reported their findings with regard to the stresses in the bone cement. As the stem material becomes more flexible, the stress distribution in the cement becomes more uniform and the stresses tend to increase. The normal stresses in the cement in the proximal portion of the stem both laterally and medially is compressive. This study contained calculations of the stresses at the metal/bone cement interface and is discussed in more detail in the next section.

Svensson et al. (1977, 1980) have compared the results of finite element analyses with strain gauge measurements. The conclusion from the first paper was that agreement with the experimental results required a slip boundary condition between the prosthesis and cement. The second paper concluded that the three most critical stress values in the system are the tensile stress on the lateral side of the prosthesis, the tensile stress across the bone/acrylic interface, and the shear stress across the bone/acrylic interface. The authors concluded

that a meaningful extension of their work would be studies of the effect of different metal/acrylic interface conditions. This work, Svensson et al. (1980) is discussed in more detail in the next section in which calculations of interfacial stresses are considered. In contrast to other studies described above, this work determined that a heavier prosthesis would be beneficial and that no significant improvements can be obtained by varying the mechanical properties of the bone cement or metal prosthesis.

Calculations of Stresses at the Metal/Bone Cement Interface

From the viewpoint of stress analysis the term interfacial stress is an imprecise one. What actually can be calculated is the stress in the metal at the interface and the stress in the acrylic at the interface. Both of these factors are important in regard to the structural integrity of the total hip replacement. The stress in the metal will determine the number of load cycles possible before fatigue failure of the stem will occur. High levels of stress in the acrylic at the interface will lead to failure of the interface due to fatigue, overload, or as has recently been suggested, by creep of the acrylic (Gibbons and Buran, 1980).

Two studies which have been concerned with use of the finite element method to calculate the stresses in the bone cement at the interface are those of Svensson et al. (1977) and Yettram and Wright (1979). The results of these calculations for the variation along the stem length of the normal stress σ_η and the shear stress τ in the PMMA at the interface is shown in Figure III-2. The direction of the normal stress σ_η is perpendicular to the implant surface. The stress distributions are qualitatively quite similar even though differences

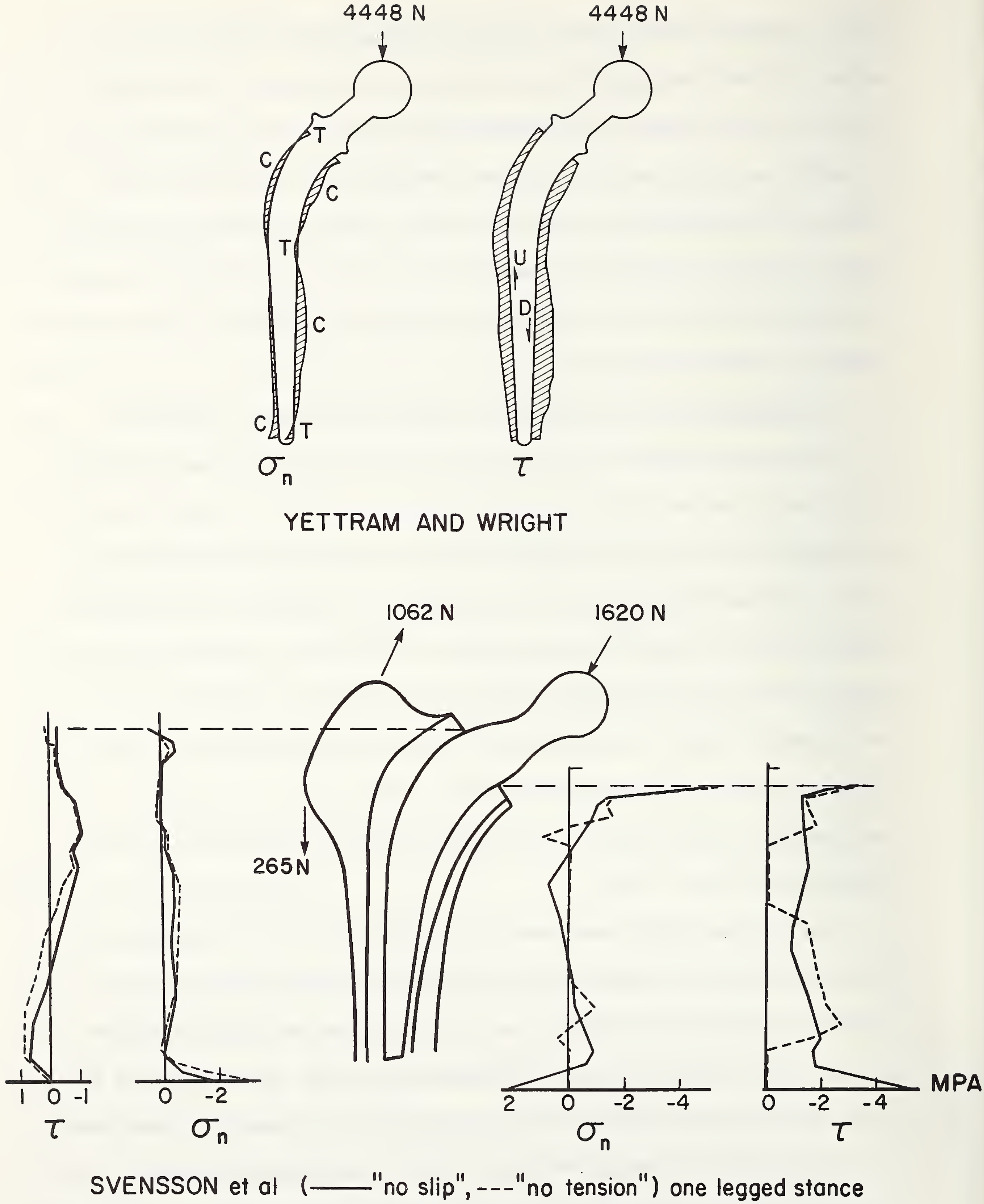

FIG. III-2 Normal Stress σ_n and Shear Stress τ in Bone Cement at the Cement/Stem Interface

exist between the two studies with respect to prosthesis shape, assumed boundary conditions, and the forces applied by the body weight and muscle tractions.

If the assumption is made that the interface will fail where the acrylic is subjected to the highest shear stresses or the highest tensile stresses, these studies would indicate that interface failure would more likely occur (a) along the medial surface than the lateral and (b) at the distal end rather than the proximal.

Interestingly, as described in Chapter IV, the clinical observations are exactly the opposite. A possible explanation for the discrepancy is that the finite element analyses do not account for the anterior-posterior transverse forces in the joint (see Chapter IV). These transverse forces produce torsional loading of the interface which probably should also be considered in calculations of the stress state of the acrylic at the interface.

Chapter IV

CONCEPTS FOR IMPROVEMENT OF STRESS ANALYSES OF TOTAL HIP IMPLANTS

This chapter briefly reviews the literature pertaining to failures of hip implants which are observed by clinicians. Although there are a great many causes of failures of total hip replacements, one important concern is the integrity of the metal/bone cement interface. As discussed by Markolf and Amstutz (1976) lack of fixation of the stem and the cement at the interface can cause a stress state in the stem which could ultimately lead to fracture of the device. The lack of correspondence between the analyses of stress at the interface given in Chapter III and the actual positions of prevalent interface failure is examined. The probable cause for the discrepancy is seen to be the neglect of anterior-posterior forces in the analysis. This would indicate that the behavior of metal/bone cement interfaces under conditions of torsional loading is an area which should be further investigated in the future.

Clinical Observations of Failure Modes of Hip Implants

A number of studies have described observations of fractures of femoral stem components (Carlsson et al., 1977), (Galante, 1980), (Collis, 1977). However, the majority of total hip replacement procedures which are performed are successful and, of those which are not, only a portion of them are failures due to biomaterials or biomechanics related causes. Nonetheless failures of the latter type are a significant problem, and it is important to understand why they occur.

Statistics in regard to failure frequencies of implants are far from complete, but a recent study by Nolan et al. (1975) puts the

matter in perspective. In a study of 3,204 total hip replacements, 3.9 percent required reoperation after 2 to 5 years. The complications which necessitated the revision were (from most to least frequent): infection, dislocations, trochanteric problems, ectopic bone, and loosening of the femoral prosthesis.

Loosening of the femoral stem component seems to be often associated with biomaterials or biomechanics related hip implant failures. Other factors also often implicated are lack of support at the calcar region and varus orientation (Galante, 1980). Loosening can occur at either the metal/bone cement interface or the bone/bone cement interface, although the latter problem has received more attention in the literature and can cause considerable pain to patients.

In a recent study by Gruen and Amstutz (1977) in which failure modes of total hip replacements were investigated, the incidence of loosening (as determined by the presence of radiolucent lines in radiographs) of the two interfaces was about equal: 10.3 percent at the metal/bone cement interface and 11.1 percent at the bone/bone cement interface. Failure as defined in their study represented a failure in the mechanical integrity of the total hip replacement and not clinical failure which is characterized by pain and restrictions in function, motion and muscle power.

In the work of Gruen and Amstutz it was shown that loosening at the metal/bone cement interface does not occur with equal probability at all points on the surface of the implant. One hundred percent of the failures occurred at the proximal end and greater than 90 percent of these occurred at the proximal lateral surface. As was previously pointed out, these clinical observations are in sharp contrast to the

predictions of the finite element studies described in the last section of Chapter III. Those investigations considered the total hip replacement to be loaded only by forces whose lines of action lie in the medial-lateral plane. Transverse forces acting in an anterior-posterior direction (which would create torsion) were neglected. Actually, as is considered in the next section, these forces can have significant magnitudes and improvements in theoretical and experimental stress analyses could certainly be made if they were not neglected.

Joint Loads

The hip is a very highly loaded joint. Estimates of the peak force which is present in the hip over the course of the walking cycle range up to six times body weight (Williams and Svensson, 1968). Measurements with instrumented femoral prostheses (Rydell, 1965), (English and Kilvington, 1979) have shown 2.5 times body weight to be present.

Of particular interest to the subject of this report are the magnitudes of the vector components of this force. This topic has been considered by Berme and Paul (1979). Their results show the components of force to vary over the walking cycle as shown in Figure IV-1. The x, y, and z axis system used is the same as that which appears in Figure III-1.

The stress analyses of Chapter III assume that only force components F_y and F_z are present, yet, as shown in Figure IV-1, the peak F_x force is about as large as the others. This would mean that a significant twisting moment would be produced about axis $\delta\delta$ (which is taken to be a principal axis of inertia of the stem in the y z plane, see Figure III-1) due to application of an anterior-posterior force at the

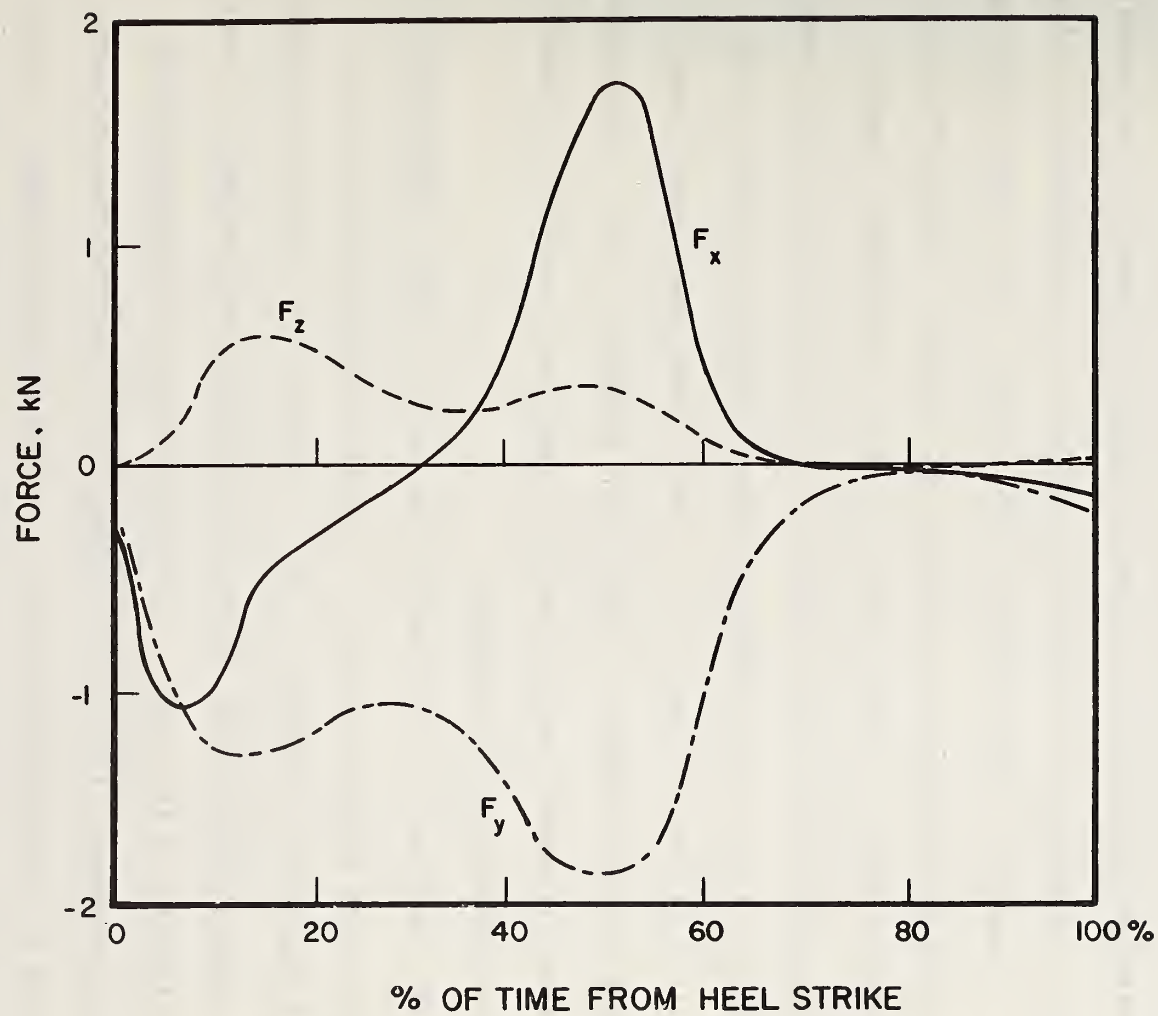

FIG. IV-1 Variation of Joint Force Components with Time During the Walking Cycle

ball of the femoral component. This would mean that the bone cement/ metal interface experiences a significant torsional load during a considerable portion of the walking cycle.

It appears that insufficient attention has been paid to the biomechanical significance of this transverse force with respect to its effect on the hip joint either in regard to trauma of the natural joint or the behavior of artificial implants. The biomechanics literature is apparently quite sparse in this respect. The search which was pursued here found only one reference to torsional loading; Mizrahi et al. (1979) showed that the shearing stresses in the metal implant corresponding to torsional loading are greatest at the proximal end of prosthesis (i.e., in the area most often observed clinically to be prone to interface failure). Also, they showed that these stresses increase with the degree of flexion and abduction (i.e., at anatomical positions different from the upright stance and for which the anterior-posterior force would be larger).

The importance of considering torsion for better understanding of the hip joint (whether natural or totally replaced) can be inferred from a few clinical studies as well. Wroblewski (1980) has suggested that several types of hip injuries and diseases are related to transverse loading of the joint. In another study of fractured femoral stems (Wroblewski, 1979), the findings indicated that the fractures were due to the combination of torsion and bending stresses. Carlsson et al. (1977) in examinations of a small number of fractured femoral stems also reports evidence of torsional deformation.

The information considered above implies that torsional loading of metal/bone cement interfaces could exert a significant influence on

their failure. Since little, if any, research has been done on the torsional characteristics of the metal/bone cement interface, experimental studies in this area were initiated. These are described in Chapter V.

Chapter V

CHARACTERIZATION AND TESTING OF METAL/BONE CEMENT INTERFACES

Initially it was thought that ASTM standard tests could furnish guidance as to appropriate testing methods for determining the strength of metal/bone cement interfaces. As is discussed below, this turned out not to be the case. The literature pertaining to mechanical testing of bone cement and bone cement/metal interfaces was then reviewed. This research is described in this chapter. Since there have been no previous studies of the behavior of the interface in response to torsional loading (a failure mode which, as previously discussed, could be important for lack of success of total hip replacements in some cases), experimental pilot studies to devise a torsion test for the interface were initiated. The results are described here in Chapter V.

Interfacial Bond Strength Tests

A number of different testing methods to determine the strengths of metal to adhesive bonds have been sufficiently well characterized that they have become ASTM Standard Tests. Among these are ASTM D1002-72 (1978) Strength Properties of Adhesives in Shear by Tension Loading, D1062-78 Cleavage Strength of Metal-to-Metal Adhesive Bonds, D2293-69 Creep Properties of Adhesives in Shear by Compression Loading, D3433-75 Fracture Strength in Cleavage of Adhesives in Bonded Joints, D3658-78 Determining the Torque Strength of Ultraviolet Light-Cured Glass/Metal Adhesive Joints, D2095-72 Tensile Strength of Adhesives by Means of Bar and Rod Specimens, D2182 Strength Properties of Metal-to-Metal Adhesives by Compression Loading, and E229-70 Shear Strength and Shear Modulus of Structural Adhesives. The modes of loading involved in some of these tests are shown in Figure V-1.

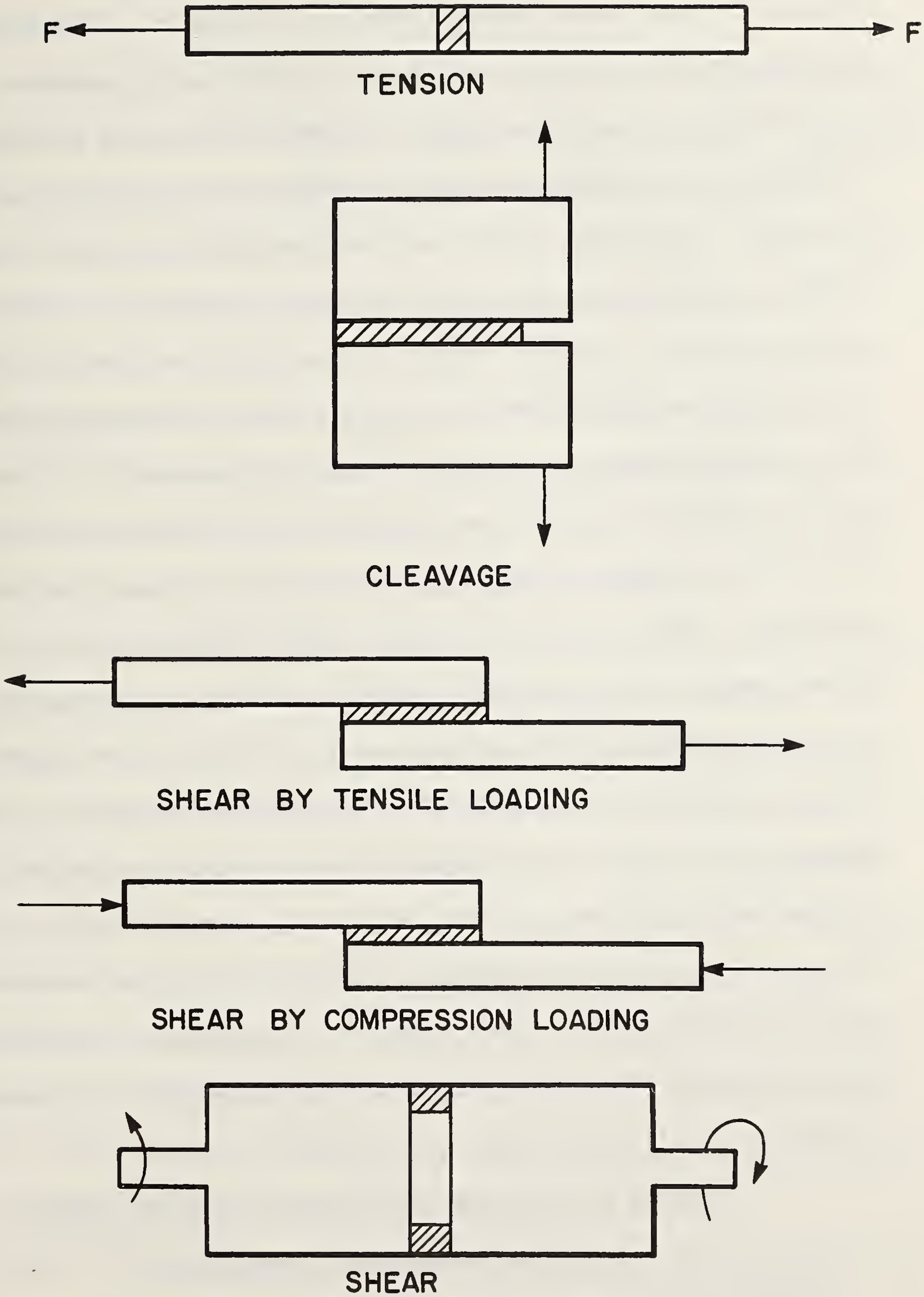

FIG. V-1 Loadinq Modes Employed for ASTM Standard Tests of Strength Properties of Adhesives

The applicability of tests suited for testing adhesives to evaluation of the bone cement/metal interface is uncertain. First of all bone cement does not function like, or only like, an adhesive. It is true that bone cement does bond to a metal surface via a mechanical interlocking mechanism and (like an adhesive) does chemically bond to the metal. The latter effect has been demonstrated by Keller et al. (1980) and others who found that different materials with the same surface finish or the same material given different chemical treatments of the surface have different strengths. Bone cement also functions as a grouting material, however. A grouting material is a space filling substance (such as mortar used to fill chinks or cracks). Thus unlike an adhesive which is a planar layer between two massive materials, the bone cement has significant thickness itself. Although a bone cement layer could be prepared to conform to the geometry of the adhesive tests, it is quite possible that the polymerization process could be altered such that the mechanical properties of the cement (and therefore the strength of the interface) would not be representative of those of bone cement when prepared in bulk. Another limitation of using the ASTM tests is that there is no standard for torsion testing of interfaces in which one component is enclosed by the other, which is probably the most meaningful test with regard to similarity to clinically observed failure.

Studies of the Mechanical Behavior of Bone Cement and Bone Cement/Metal Interfaces

The polymethylmethacrylate bone cements which are used for the total hip replacement have been extensively investigated biomaterials. The literature which has been generated will be briefly reviewed here;

only the papers dealing with interfacial strengths will be examined in more detail.

Studies by Kusy (1978), Haas et al. (1975), and Edwards and Thomasz (1981) have used various chemical, physical, and microscopic techniques to characterize and evaluate the chemical composition, degree of polymerization, porosity, and other microscopic features of the cement. The latter two investigations were also concerned with the curing characteristics of the material. Other studies have also been concerned with the curing characteristics of the cement *in situ*. Sih, Connelly, and Berman (1980) studied the temperature-time history of *in vitro* samples of setting bone cement. Ahmed et al. (1979) calculated the thermal stresses produced as bone cement solidifies.

Other research has dealt with effects (both positive and negative) on the mechanical properties of PMMA bone cement which results when different components are added to the basic composition for various purposes. Pilliar et al. (1976) and Saha and Kraay (1979) examined the influence of fiber reinforcing materials. Combs and Greenwald (1979) studied the effects of barium sulfate. Bayne et al. (1975) and Rijke et al. (1977) investigated the dependence of properties on porosity in the bone cement. Lautenschlager, Marshall, et al. (1976); Lautenschlager, Jacobs et al. (1976); Nelson et al. (1978), and Moran et al. (1979) have determined the influence of antibiotic additions on the mechanical behavior.

Many other studies of the mechanical behavior of bone cement have been conducted. Holm (1977) measured flexural strength and Young's modulus. Lee et al. (1977) performed bending, compression, and tensile tests. Freitag and Cannon (1977) studied the fatigue behavior of bone

cement. A number of investigations have been pursued in which a fracture mechanics approach has been used to study bone cement. These include (Beaumont, 1977), (Beaumont, 1979), (Beaumont and Young, 1975a), (Beaumont and Young, 1975b), (Owen and Beaumont, 1979), (Sih and Berman, 1980), (Robinson et al., 1981), and (Kusy, 1978).

Two studies have focused on the effect of the in vivo environment on the mechanical behavior of bone cement. Rostoker et al. (1979) tested samples of PMMA bone cement which had been implanted in rabbits and found a statistically significant decrease in fracture stress after 26 months. Gibbons and Buran (1980) examined bone cement samples which had been retrieved from total hip replacements. Their findings were plastic deformation resulting from creep, abrasive wear of the cement, and cracks radiating from the corners of the medial edge.

Several investigations have been conducted which tested the strength of the bone cement/metal interfacial bond. These will be discussed in detail. Beaumont and Young (1977) performed push-through tests on stainless steel cylinders embedded in bone cement. Failure occurred either by shear fracture of the cement or disruption of the interface. The load-displacement curves exhibited a linear portion up to a maximum load P_i and then a slowly decreasing load with further displacement in a saw-tooth pattern corresponding to stick-slip failure. The interface shear strength was defined as:

$$\tau_{is} = P_i/\pi d L_e \qquad (1)$$

where d is the rod diameter and L_e is the length embedded in acrylic.

Various types of surface conditions of the rod were examined. τ_{is} for a 1 mm deep thread was 45 MPa (which according to the investigators corresponds to the shear strength of the cement). τ_{is} was

observed to be insensitive to surface finish below center-line averages (determined with a Talysurf measuring device) of 8 μm. For these surfaces τ_{is} was seen to be 9.2 ± 3 MPa. The presence of water at the interface had a pronounced adverse effect on τ_{is} and decreased it from 9.2 to 4.7 MPa.

In a study which used a somewhat similar approach, Greenwald and Wilde (1974), measured τ_{is} to be 0.207 MPa for bone cement/high density polyethylene interfaces, between 2.12 and 3.35 MPa for cancellous bone/bone cement interfaces, and 1.35 MPa for stainless steel/bone cement interfaces. The investigators speculated that knurling or roughening the surface of the metal might enhance the bond strength.

There was quite a descrepancy between the investigations of Beaumont and Young; and Greenwald and Wilde with regard to the shear strength of the bone cement metal interface. Perhaps the difference lies in the length of the samples used. The influence of sample length on test results was considered in a more refined treatment of the problem by Beaumont and Plumpton (1977). They performed pull out tests on bone cement/metal interfaces and analyzed their results in a more complete manner than previously using shear-lag theory [see Greszczuk (1969) for a description of shear-lag analysis]. The experimental results indicated that the average shear stress τ_{av} (identical to τ_{is} above) increases as L_e decreases.

The reason for this effect is that the stress at the interface in a pull-out test can be shown by a more complete analysis to vary along the length. The stress is not constant as implied by equation 1 but rather is a function of distance x along the length away from the point of application of the force. The functional dependence is given below (Beaumont and Plumpton, 1977):

$$\tau(x) = \frac{P_i \alpha}{2\pi r}(\sinh \alpha x - \coth \alpha L_e \cosh \alpha x) \qquad (2)$$

where

$$\alpha = \left[\frac{2G_i}{b_i r E_f}\right]^{1/2} \qquad (3)$$

In the above equation r is the radius of the rod, E_f is its Young's modulus, b_i is the interface thickness (which can be estimated from the surface roughness), and G_i is the interface shear modulus. τ_{max}, the maximum interfacial stress, occurs at x = 0 and is given by:

$$\frac{\tau_{max}}{\tau_{av}} = \alpha L_e \coth \alpha L_e$$

As $L_e \to 0$, $\tau_{av} \to \tau_{max}$. Thus, measuring τ_{av} vs L_e and extrapolating to $L_e = 0$ determines τ_{max}. For any L_e, τ_{max}/τ_{av} can then be determined from a pull out test. With this information α can be obtained, from which G_i can be extracted.

For dry interfaces and interfaces in contact with blood, interface weakening was also observed in their study due to the fluid contact. τ_{max} was found to be 18 and 10 MPa respectively. G_i was observed to be 10^3 and 10^2 MPa respectively. Beaumont and Plumpton cite the finite element studies of McNeice and Amstutz (1975) which find stresses at the interface of 16 ± 2 MPa and concluded that interface failure could occur early in the life of hip prostheses.

Shear-lag analysis seems to offer promise to assist in the data reduction for the experimental torsion studies of the type for which

preliminary results are described in the next section. It may also offer insight into the torsional stresses present in hip implants due to the action of the anterior-posterior force which is present during the walking cycle. These possibilities should be futher investigated.

Another study of the bond strength between bone cement and implant material has been conducted by Raab et al. (1981). They measured interfacial shear strengths by push through tests for bone cement/metal interfaces and by shear through compression loading tests for UHMWPE/bone cement interfaces. The metals investigated were Co-Cr-Mo, Ti-6Al-4V, and 316 LVM. Tests were performed at a strain rate of 1.3 x 10^{-3} s^{-1} since it was observed that interface strength and elastic modulus increased with strain rate above a plateau region which ended at about 10^{-1} s^{-1}. Tests were performed in both Ringers solution at 37 °C and in a dry environment. Interface stresses were calculated using finite element techniques. For pullout tests their calculations indicated that the stress is a function of the length, a result similar to the work of Beaumont and Plumpton cited above. Along a radial direction from the cylinder the stresses are highest at the metal surface and decline with increasing distance. A fracture mechanics approach was used for tests with other specimens to find the crack extension force. In still other studies Raab et al. measured the fatigue behavior of interfaces. S-N curves which appear to exhibit endurance limit type behavior were found.

Most interesting from the standpoint of this report were their studies which measured the interface strengths for the different metal/bone cement surfaces after various times of immersion in saline solution. The results are shown in Table V-1. The different metals

TABLE V-1

SHEAR STRENGTHS OF BONE CEMENT/MATERIAL INTERFACES FOR VARIOUS METALS AND UHMWPE[a], τ_{is}(MPa)

Interface	Dry	30 days (saline)	60 days (saline)	Dry (conditioned)
Co-Cr-Mo/B.C.[b]	6.9 ± 0.7	5.3 ± 0.8	5.3 ± 1.0	6.0 ± 0.6
Ti-6Al-4V/B.C.	12.5 ± 1.8	6.3 ± 0.6	6.7 ± 1.3	12.2 ± 1.6
316L VM/B.C.	11.2 ± 1.9	6.2 ± 0.4	6.4 ± 1.1	8.2 ± 1.4
UHMWPE/B.C.	2.3 ± 0.4	0.5 ± 0.3	0.4 ± 0.2	1.6 ± 0.2

[a] after Raab et al (1981)

[b] B.C. - bone cement

showed a considerable range of interfacial shear strengths when tested dry. Co-Cr-Mo/bone cement interfaces were less than 60 percent as strong as Ti-6Al-4V/bone cement interfaces. The surface finishes on these materials were 3.0 μm or better. This value lies within the range where τ_{is} was observed by Beaumont and Young (1977) to not depend upon surface finish. The metals were prepared according to ASTM standard F86, Standard Recommended Practice for Surface Preparation and Marking of Metallic Surgical Implants. They were grit blasted and passivated in HNO_3 24 h before testing and autoclaved 2 h before testing. Conditioning surfaces by exposure to laboratory air for two weeks prior to testing diminished interfacial strength in 316L and UHMWPE. In all cases the metal/bone cement bonds were much stronger that the UHMWPE/bone cement bonds. Differences in τ_{is} between metals diminished with time of saline exposure as 316L and Ti-6-4/bone cement bonds deteriorated in strength. The authors concluded that the strength deterioration is an interfacial effect rather than a bulk effect in the bone cement (since other tests on bone cement itself did not show degradation of its properties with comparable saline exposures). They attributed the degradation of surfaces due to conditioning by exposure to laboratory air to formation of surface oxide.

It can be presumed that chemical interaction or bonding between the two materials of the interface could occur since different materials with similar surface finishes have different τ_{is}. Similar findings were made by Keller et al. (1980).

They also studied the variables which affect the strength properties of bone cement/metal interfaces. The method used in their investigation was to measure the interfacial bond strength in tension σ_i. This test has the disadvantage that the failure which is produced

occurs both at the interface and also within the acrylic itself so that some parts of the surface are covered with cement. Keller et al. studied the influence on σ_i of interface formation time (i.f.t.), i.e., the time after the onset of mixing that contact was made between the metal and bone cement, and curing time (the time elapsed between sample preparation and mechanical testing). The materials they investigated were 316L, Ti-6Al-4V, and Co-Cr-Mo. In one series of tests the samples used were polished to 0.1 μm and subsequently electropolished and prepassivated. In another series the samples had a 15 μm finish.

The results of this investigation showed that differences in σ_i resulted for different cure times and interface formation times. The various metals behaved differently, however, so that no general trends can be observed in the data, except that late i.f.t.'s lead to inferior properties. The results indicated that, in contrast to the observation of Raab et al. (see Table V-1), 316L/bone cement interfaces were stronger than the other interfaces (6.5-11.7MPa for 316L depending upon cure time and i.f.t. as compared to 5.1-9.7MPa for Co-Cr-Mo and 1.9 to 8.4 MPa for Ti-6Al-4V). This effect was more pronounced at 1 h than after 1 week. Thus the work of Keller et al. also indicates more similar behavior between metals as time goes on. Somewhat surprisingly, the rougher surfaces had lower values of σ_i than the prepassivated surfaces. This could mean that roughness is not a factor below 15 μm but that passivation treatments promote chemical interactions and bonding between the metal and acrylic. Also, as suggested by these investigators, roughness might lead to air bubble entrapment at the surface.

For 316L, tests in another series of samples were run in 37 °C Ringer's solution. Like Raab et al., reduction in σ_i was noted in this study as well. The effect was most pronounced for late i.f.t.'s when the doughy mass of bone cement has high viscosity and would be expected to have considerable porosity at the interface leading to water contact with the interface by sorption processes.

Experimental Studies

Since the literature search previously described led to the conclusion that the response of the bone cement/metal interface to torsional loading appears to be a question which should be investigated to obtain further insight into failures of total hip replacements, a series of experiments was pursued in this area. The specimen used for the mechanical tests is shown at the top of Figure V-2. A cylindrical metal rod of 0.635 cm diameter was embedded in a rectangular prism of bone cement such that their axes were coincident. Torque was applied to the free end of the rod which tended to twist the metal surface relative to the acrylic surface at the interface. This type of sample geometry was selected over that used in ASTM Standard E229-70 Shear Strength and Shear Modulus of Structural Adhesives (see the bottom of Figure V-1). It was thought that because the metal is surrounded by the acrylic this sample represents a closer approximation to the geometry of the total hip replacement.

Ten samples of this type were prepared. The manufacturer's instructions to the surgeon were followed in the preparation of the bone cement (Howmedica Surgical Simplex P). The powder (consisting of PMMA and methyl methacrylate-styrene copolymer with or without barium sulfate added for radiopacity) and methyl methacrylate monomer were mixed in the recommended 2 g to 1 ml ratio. The contents were stirred

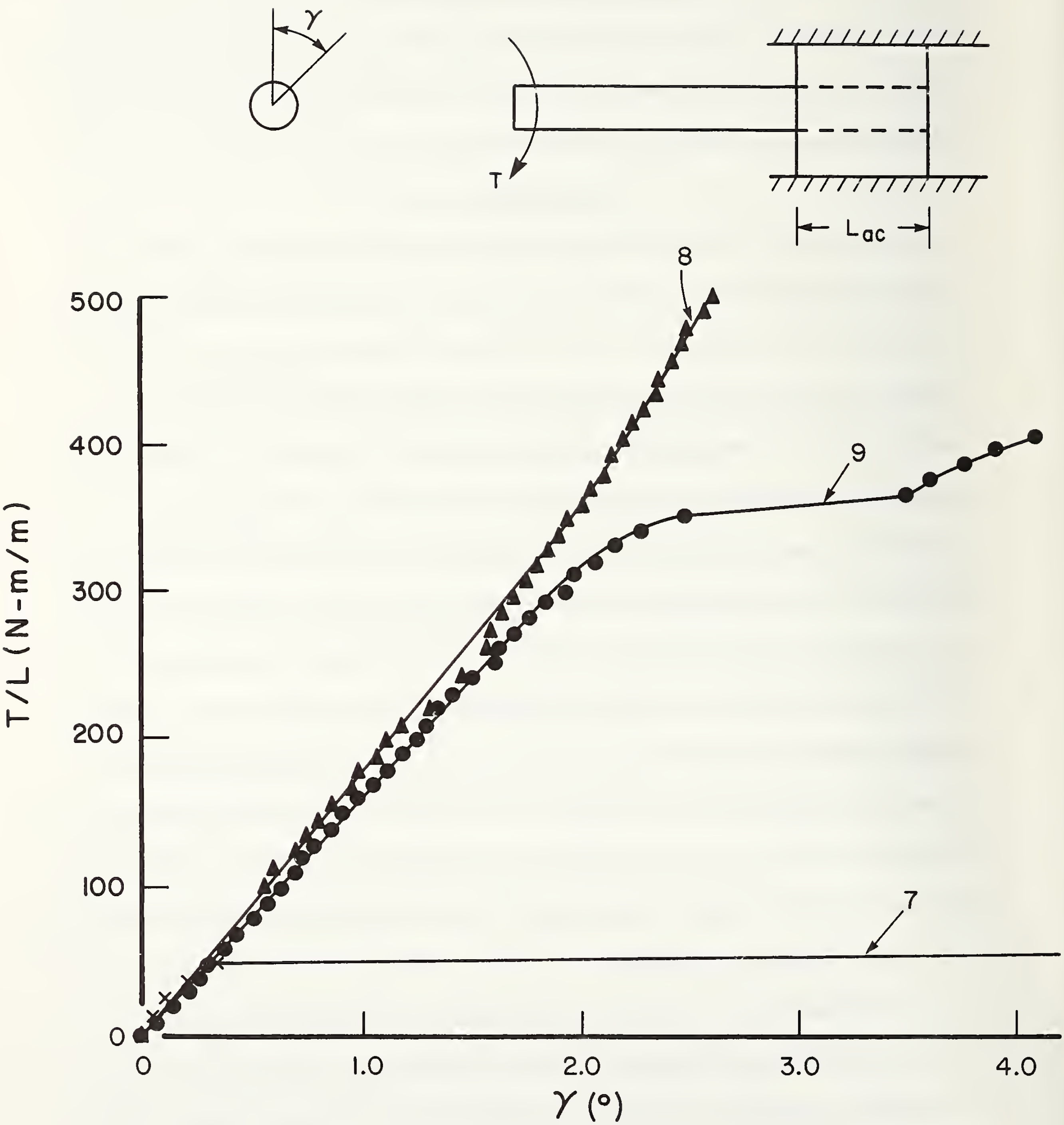

FIG. V-2 Length-normalized Torque T/L_{ac} vs. Twist Angle γ for Interfaces 7, 8 and 9.

for two minutes and the doughy mass was kneaded approximately two minutes longer, after which the bone cement was brought into contact with the metal rod. Thus the interface formation times ranged from 4 to 4-1/2 minutes.

The metal samples were 316L stainless steel polished to a 15 μm finish. Before the interface was formed the rods were washed in a detergent solution, rinsed, and ultrasonically cleaned in ethanol. The interface was formed through use of a Teflon mold. The metal sample was aligned parallel to the axis of the mold. The assembly was equipped with a Teflon plunger to compress the bone cement as it was hardening to promote better contact with the metal and reduce porosity, thus producing higher quality interface formation. Some interfaces were formed using a small weight for pressure on the plunger, as was done by Keller et al. (1980) to simulate a currently used technique in total hip arthroplasty. Other interfaces were formed by applying as much pressure as possible by hand to the plunger for 5-1/2 minutes as the cement hardened.

It was observed that the interfaces made with the low pressure were poorly formed and fragile compared to those made with the high pressure. The decision was made to form the interfaces which were to be mechanically tested by the higher pressure technique. It was felt that, although the higher pressure method less closely simulated the clinical situation, this would not be a significant disadvantage because so many other variables also affect interface formation _in vivo_ in the operating theater. The higher pressure technique seemed to offer the advantage of producing a more reproducible, higher quality interface with a greater true surface area of contact between the

metal and bone cement. Reproducibility is important for studies where different surface treatments of the metal (e.g., polished, passivated, roughened, scribed, etc.) are examined with respect to their influence on the strength of the metal/bone cement interface.

Interfaces which were formed with the high pressure method were tested in torsion. The testing apparatus used was a modification of a torsion machine which has been used in corrosion fatigue studies as described by Imam et al. (1979). In its modified form the machine comprised a dead weight loading system. A cable, pulley, loading platform, and weights were used to apply a torque to a shaft which could be calculated from the weight W and geometrical configuration. At the other end of the shaft was a grip attached to the free end of the 316L rod. The end of the rod embedded in acrylic was held in a grip which fixed the acrylic block rigidly in position.

The twist angle γ was measured by determining the change in position of a laser beam which was reflected from a mirror whose axis was coincident with that of the shaft and which was attached at the end where the torque was applied. The twist angle is produced predominantly by distortion of the acrylic block as torque is applied to the rod embedded within it or by relative movement of the two components at the interface. The metal rod, grips, and shaft which applies the torque are presumably virtually rigid in comparison.

Preliminary experiments were performed in which the W vs γ response was measured for samples in which the length of the rod embedded in the acrylic L_{ac} ranged from about 1.25 cm to 5 cm. These experiments demonstrated that there was a linear relationship between W and γ which was reproducible upon loading, unloading, and subsequent

reloading. They also indicated that smaller values of L_{ac} would have to be used in order to stress the interface beyond the region of linear elastic response without exceeding the maximum loading capacity of the dead weight system.

Results from three tests in which torque T vs twist angle γ was determined for samples in which L_{ac} was under 0.635 cm are shown in Figure V-2. The torque is normalized by dividing by L_{ac} to afford a fairer comparison of the different tests. Several conclusions may be drawn from this data. The curves obtained experimentally validated the concept of using a sample of the geometry shown at the top of Figure V-2. That is, "failure" of the interface itself was observed rather than fracture of the acrylic which can occur sometimes with other types of geometries. "Yielding" at the interface seems to occur when the T vs γ curve departs from linearity. The twist which results when the T vs γ curve becomes nonlinear is permanent deformation. This could be easily seen at high twist angles when the sample had been visibly rotated in the acrylic.

Quantitatively the elastic portion of the T vs γ curve was fairly reproducible for the three tests. Table V-2 gives the values observed for the proportionality constant G" between T and γ for the linear portion of the curve governed by the equation $T = G''\gamma$. As yet, an analytical treatment of how to calculate G" from the sample geometry and the shear moduli of the metal and bone cement has not been carried out. However, undoubtedly the effective stiffness is increased as the length of metal embedded in acrylic L_{ac} increases. As shown in the table, when G" is normalized to L_{ac} the standard deviation was within 20 percent of the mean.

TABLE V-2

EXPERIMENTAL VALUES OF PROPORTIONALITY CONSTANT G"
BETWEEN TORQUE AND TWIST ANGLE

Interface	G" (N-m/degree)	L_{ac} (cm)	G''/L_{ac} (N/degree)
7	0.691	0.47	147.0
8	1.134	0.56	202.5
9	0.837	0.58	144.3
Mean			164.6 ± 32.9

The value of torque required to produce yielding at the interface T_y, could easily be quantitatively determined from the T vs γ curve. Although this point does not correspond to complete fracture of the interface in which the two materials are separated (since the torque does not go to zero) it probably represents a stress level beyond which the interface should not be stressed in order to avoid subsequent damage and can be taken to represent a practical criterion for failure of the interface.

Time dependent effects were observed to be associated with yielding. When a weight increment was applied in the region beyond the linear region, it would take several seconds for significant twist to occur but the twist would gradually increase with time at a decreasing rate until it ceased. This behavior seems similar to a creep phenomenon.

The T_y observed in the tests shown in Figure V-2 varied widely. T_y for interface 7 was about 0.23 N-m, for interface 9 was about 2 N-m, and for interface 8 was over 2.8 N-m and could not be observed within the loading capacity of the torsion testing machine. Part of this variation is to be expected. Keller et al. (1980) in extensive mechanical studies of metal/bone cement junctions did observe standard deviations which exceeded 50 percent of the mean interface strength. Interfaces 8 and 9 could have a T_y range compatible with this figure. However, interface 7 failed at a much lower value. Presumably the reason lies with differences in the preparation of the samples. The cure time for interface 7 was 9 days compared to 7 days for interfaces 8 and 9. The total amount of bone cement prepared (from which the mass used to form the acrylic block was taken) for interface 7 was

half of that used for 8 and 9. The properties and quality of the bone cement does depend somewhat upon the quantity which is polymerized. Also, interfaces 8 and 9 were formed from the radiopaque bone cement formulation whereas interface 7 was formed from the plain cement composition. The material used for interface 7 had been manufactured about 6 years prior to that used for interfaces 8 and 9.

In future tests it is probable that the variability could be reduced by more uniform preparation of the interface, through use of hydraulically applied pressure to the mold, and mixing of identical amounts of material. Also, equal cure times should be employed for all samples. Testing with more sophisticated mechanical testing machines (than that used here) which can measure the time dependent behavior of the interface could also be of assistance in this regard.

Chapter VI

SUMMARY AND RECOMMENDATIONS

The main conclusions to be drawn from this literature search can be summarized as follows:

1. Until biomaterials exist which match bone in its elastic properties and are also otherwise suitable for implantation, stress shielding of bone in the vicinity of implants will occur. Bone resorption is a possible consequence. The area of the total hip replacement most likely to be adversely affected is the calcar femorale. Calcar resorption can create high stress situations in the implant itself leading to its eventual failure. Implant designs which minimize stress shielding are to be preferred.

2. Most of the experimental and theoretical techniques which are used by stress analysts have been applied to bone and many problems of importance to hip implants have been examined. The unique conditions present within the body create uncertainties with regard to these analyses. The theoretical analyses are particularly sensitive to uncertain boundary conditions, irregular geometry, and nonuniformity in elastic properties.

3. Few studies have been conducted of the stresses present _in vivo_ at the metal/bone cement interface. The theoretical studies which have been done do not show maximal stress patterns in the areas where clinical failures are most likely seen.

4. The reason that there is a discrepancy between theoretical calculations and clinically observed failures is probably because of the neglect in the computations of the anterior-posterior joint forces which are present during the walking cycle.

5. Since failures of the bond between metal and bone cement occur in practice and can lead to failures of the device itself, development of testing methods to determine the bond strength are important. ASTM tests which are used for measurement of properties of adhesives are not suitable.

6. Many studies of the physical and mechanical properties of bone cement have been conducted. Studies of the strengths of the bone cement/metal interface in tension and shear have been made. The strength is dependent upon many variables including metal surface preparation and roughness, cure time, interface formation time, composition of the metal, air or Ringers solution environment, time between preparation of the metal surface and interface formation, etc.

7. Since no studies of the bone cement/metal interface have been done when torsional forces act at the interface and the literature search indicated that torsion could be most important for interface failure, preliminary experiments were performed. The results indicated that a sample in which a cylindrical rod was embedded in an acrylic block could allow an interfacial yield strength to be measured.

Based upon the results of this research the following recommendations can be made:

1. Further investigation of the importance of torsional loading to the stresses developed at bone cement/metal interfaces should be pursued to gain increased insight into service failures of total hip replacements. Methods such as shear-lag analysis or three dimensional finite element analysis should be employed for this purpose.

2. Further study should be pursued of the optimum test sample geometry for torsion specimens. Refinements should be made in the manner in which interfacial strength and the proportionality constant between torque and twist are determined from the experimental data.

3. Since time dependent effects were observed in this study and creep has been observed in failed bone cement, further mechanical tests to be pursued should be performed with a testing machine in which strain rate can be controlled. Also since both a torsional force and a downward force exist _in vivo_ during the walking cycle, some tests with a machine with biaxial capabilities should be performed.

4. Systematic studies of ways to improve the strengths of bone cement/metal interfaces should be investigated. The common implant metals and various types of surface finish (such as polished, roughened, porous, passivated, knurled, etc.) should be studied. Preparation techniques for the interface should be carefully controlled.

5. Scanning electron microscope studies of the interface between bone cement and implant metals should be pursued to determine the true contact areas between the two materials. This research should be correlated with mechanical testing data to determine the intrinsic strength of the interface based upon actual contact area. This would allow results of laboratory tests of interfacial strength to be extrapolated to the clinical situation if SEM studies of retrieved bone cement samples were done to determine the contact area present _in vivo_ at the metal/bone cement interface.

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Part III

SURFACE PREPARATION AND CORROSION BEHAVIOR OF TITANIUM ALLOYS FOR SURGICAL IMPLANTS

A. C. Fraker, A. W. Ruff, P. Sung, A. C. Van Orden
and K. M. Speck

SURFACE PREPARATION AND CORROSION BEHAVIOR OF TITANIUM ALLOYS FOR SURGICAL IMPLANTS

A. C. Fraker*, A. W. Ruff*, P. Sung***, A. C. Van Orden*
and K. M. Speck**

*National Bureau of Standards, Washington, DC 20234
**Uniformed Services University Medical School,
Bethesda, MD 20016
***Food and Drug Administration, Silver Spring, MD 20857

ABSTRACT

Surface preparation and corrosion behavior of titanium alloys were studied; both topics deal with the formation of surface oxide films. When metals are prepared for surgical implant use, an effort is made to produce an optimum surface. Effects of the surface treatments on surface morphology, surface film composition, and structure are shown in transmission electron micrographs and electron diffraction patterns. Roughened surfaces were produced and surface films of TiO and/or TiO_2 occurred on some specimens. NaO·xTiO_2 occurred on the specimens washed in NaOH solution. Open circuit potential vs time curves have been measured to show some electrochemical effects of various surface treatments. Results show that titanium alloys immersed in Hanks' physiological solution reach the same final open circuit potential after approximately two weeks' exposure regardless of prior surface treatment. Measurements of the anodic polarization behavior of titanium alloys and other surgical implant alloys show the effects of alloy composition and testing solution on the passive region and breakdown potentials of these materials. In general, for titanium alloys, the differences are not great but the presence of nickel results in a significant lowering of the breakdown

potential. The materials studied were titanium, Ti-6Al-4V, Ti-Ni (memory alloy), Ti-13Cu-4Ni and Ti-4.5Al-5Mo-1.5Cr. In all cases titanium materials are more corrosion resistant in Hanks' solution than the Co-Cr-Mo alloys, 316L stainless steel or the Co-Ni-Cr alloy. Repassivation measurements show the rapid formation and the high degree of stability of the protective film on the titanium and titanium alloys.

SURFACE PREPARATION AND CORROSION BEHAVIOR OF TITANIUM ALLOYS FOR SURGICAL IMPLANTS

Introduction

The purpose of this paper is to present the effects of surface preparation on electrochemical behavior of titanium alloys in a simulated physiological environment and to show the general corrosion behavior and repassivation characteristics of titanium alloys in physiological solutions. While these topics are related and both deal with the formation of surface oxide films, the specifics of each are presented separately.

Titanium alloys have a high corrosion resistance in saline solutions which are typical of physiological environments. This has been the finding of Hoar and Mears [1] and a number of other research investigators [2-8]. Solar [9], in a thorough review of the corrosion resistance of titanium surgical implant alloys discussed the nature of the passive film, *in vitro* corrosion measurements, *in vivo* biocompatibility and clinical observations. This review concluded that the titanium alloys probably are the most corrosion resistant and biocompatible metals in use today.

Several investigations involving titanium alloys are reported in this paper, and all support the superior corrosion resistance of titanium alloys over other implant metals when exposed to saline solutions. These studies have dealt primarily with anodic polarization, open circuit potential and repassivation measurements. Anodic polarization curves show the current density in the passive region, the span in potential of the passive region and the breakdown potential. Repassivation studies provide a means for determining kinetics of surface film

formation and subsequent stability, and these data have shown that titanium alloys repassivate readily and have stable surface oxide films up to a potential of 2 volts. Open circuit potential measurements and transmission electron microscopy observations of surface preparation effects on titanium alloys indicate that acid or alkaline treatments cause surface roughness and affect initial surface films. Washing in boiling water or boiling saline water would be better, assuming that the material is sufficiently clean.

Part 1. The Effects of Surface Preparation

Electrochemical Measurements. Earlier work of Revie and Greene [3] showed that effects of passivating on the corrosion behavior of titanium materials in saline solution were negligible while such treatments significantly improved the corrosion behavior of 316L stainless steel. The prescribed surface treatments of ASTM F-86 for metals to be placed in the body will result in different effects for the various currently used surgical implant alloys. This can be explained partly by considering the data in table 1, taken from the "Handbook of Physics and Chemistry" [10] to show oxide solubility.

Fraker and Ruff [11] showed that titanium alloys reach the same rest potential in Hanks' physiological solution regardless of prior treatment. Some examples of this are shown in figures 1 a-e. All voltages are given in reference to the hydrogen electrode. The solid curve in figure 1a shows that the initial open circuit potential for a mechanically polished specimen immersed in Hanks' physiological solution at 4 °C is -0.10 volts (e_H) which rapidly changes to +0.14 volts. Results from a mechanically polished and steam sterilized specimen at room temperature,

Table 1. Solubility of Selected Metal Oxides [10]

Oxide		Solubility in acid (a), alcohol (al), alkaline (alk)*
TiO_2	Brookite Anatase Rutile	s H_2SO_4,alk; i a
TiO		s dil. H_2SO_4; i HNO_3
Ti_2O_3		s dil. H_2SO_4; i HNO_3, HCl
FeO		s a; i al, alk
Fe_2O_3		s HCl, H_2SO_4; sl s HNO_3
Fe_3O_4		s Conc. a; i al, eth
$Fe(OH)_2$		s a, NH_4Cl; i alk
CrO_2		s HNO_3
CrO		i dil HNO_3
Cr_2O_3		i a, alk, al
$Cr_2O_3 \cdot xH_2O$		s a, alk; sl s $NH_4(OH)$
$Co(OH)_2$		s a, NH_4 salts; i alk
CoO		s a; i al, NH_4OH
Co_2O_3		s a; i al
Co_3O_4		v sl s a; i aq. reg.

* s, soluble; v, very; sl, slightly; a, acid; alk, alkaline; al, alcohol; i, insoluble; eth, ether.

23 °C, are shown in the dashed curve and have a rest potential of +0.22 volts. All specimens for figures 1 b-e were kept at 23 °C. Figure 1b shows that a similar specimen, washed in boiling sodium chloride prior to sterilizing has an initial potential of +0.14 volts after sterilizing and a rest potential of +0.24 volts after 14 days. Figure 1c shows the open circuit potential measurements for a titanium specimen washed in 30% HNO_3 at 60 °C, steam sterilized and immersed in Hanks' solution. Here the specimen has a positive open circuit potential of +0.10 volts which levels off to +0.25 volts after 15 days. Washing titanium and titanium alloy specimens in alkaline solutions such as NaOH results in the dissolving of the surface oxide film [10]. The titanium specimen of figure 1d had this treatment and the initial open circuit potential was -0.13 volts but after 5 minutes reached 0 volts and quickly rose into the positive voltage range. This specimen eventually reached a rest potential of +0.20 volts. Figure 1e shows open circuit electrode potential measurements for titanium which was passivated in 30% HNO_3 at 60 °C and was unsterilized. Note the initial high positive potential of +0.68 which changes with time to a rest potential of +0.22 volts.

It is seen in figures 1a-e that all specimens exhibit open circuit potentials which rise and after two to four weeks fall to rest potentials ranging from +0.20 volts to +0.24 volts. The similarity in the rest potentials of the different specimens indicates that in this simulated physiological solution, the surface film on titanium materials will tend toward an equilibrium for this given environment regardless of prior treatments such as alkaline washes, acid washes or sterilization. Thicker films which would give a significantly more positive initial

open circuit electrode potential could be a source of ion release as the surface film changes to a film more stable in the given environment. Surface oxide films as a possible source for titanium ion release have been discussed previously [12]. The oxide films merit further study since titanium is highly corrosion resistant to body fluids, and it is difficult to explain titanium metal ion release into tissue surrounding an implant on any other basis.

Characterization of Surfaces and Films. Oxide film morphology and composition on titanium alloys also are affected by the type of surface treatment applied. Titanium is a metal which undergoes a series type of oxidation sequence. The surface film changes from a lower to a higher oxide as oxidation progresses and as temperatures are increased. The following phases form; $Ti + O \rightarrow Ti(O) \rightarrow Ti_6O \rightarrow Ti_3O \rightarrow Ti_2O \rightarrow TiO \rightarrow Ti_2O_3 \rightarrow Ti_3O_5 \rightarrow TiO_2$. This has been reported for gaseous oxidation of titanium by Kornilov [13] and for aqueous oxidation from TiO to TiO_2 by Fraker and Ruff [14]. If titanium has not been exposed in aqueous environments in excess of 100 °C, the surface film will be of a lower oxide type such as TiO.

The titanium oxide films are insoluble in most acids and remain protective even after an acid washing treatment. This was indicated by the positive electrode potential measurements of figure 1e after the HNO_3 passivating treatment. The titanium oxides are not stable in alkaline solutions, and the surface film resulting after treating in these solutions is a mixture of titanium oxide and the alkali metal oxide.

Titanium thin foils were prepared by electropolishing [14] in a solution of 200 ml of methanol, 115 ml of 1-butanol and 20 ml of 70 percent perchloric acid at a temperature of -40 °C to -50 °C and at a voltage

of 13 to 15 volts. The thin foils were given designated treatments and were studied using transmission electron microscopy techniques. The transmission electron micrographs of figures 2 through 6, respectively, show the pure untreated titanium, titanium after immersing in boiling water for 30 minutes, titanium after immersing in boiling NaCl solution for 30 minutes, titanium after immersing in NaOH solution for 15 minutes, and titanium after passivating in 30% HNO_3 at 60 °C for 30 minutes. All specimens except the untreated titanium were steam sterilized finally.

Electron diffraction patterns are inserted in figures 3 through 6. The presence of the oxide, TiO, after the plain water or saline water treatment is indicated by the diffuse rings. The diffraction spots are from the titanium metal. After steam sterilization, some patches of higher titanium oxides can occasionally be found but these are not predominant [7]. Immersion of titanium in alkaline solution such as 0.5N NaOH results in a combined form of surface oxide which is not as protective as any titanium oxide form. The film covering the surface in figure 5 is a form of sodium titanate, $NaO \cdot xTiO_2$. After immersing in hot HNO_3 and steam sterilizing as shown in figure 6, the resulting surface film is TiO_2, anatase. Earlier studies [15] have shown that lowering the solution pH enhances the nucleation and growth of the titanium surface film, TiO_2, anatase. It is apparent from these electron micrographs that the titanium surfaces are smoother and less affected after treatments in either plain or saline water than after acid or alkaline treatments.

Part 2. Anodic Polarization Behavior and Repassivation Studies

Anodic Polarization. Anodic polarization measurements were used to determine the passive range and the breakdown potential for a number of metals which are used currently as surgical implants. Experimental details of the procedure and composition of the materials have been given in a paper [16] describing corrosion behavior of the memory alloy, Ti-Ni and Ti-6Al-4V. All materials were implant grade as specified by ASTM designated compositions. Specimens were prepared by mechanically polishing through 0.05 μm Al_2O_3 and then were anodically polarized. The potential was increased at a stepping rate of 0.006 V/min. in Hanks' physiological solution. All potentials were measured in reference to a saturated calomel electrode. Hanks' solution was prepared [16,17] and tests were conducted at 37 °C and a pH of 7.4. Results showed that the titanium alloys tested had superior corrosion resistance over the other metals tested by a large margin. The breakdown potentials for these materials are given in table 2.

Table 2. Breakdown potentials for implant metals in Hanks' solution

METAL	BREAKDOWN POTENTIAL (VOLTS vs S.C.E.)
316L Stainless Steel	+0.2 to +0.3*
Co-Cr-Mo	+0.42
Co-Ni-Cr	+0.42
Ti-Ni	+1.14
Ti-6Al-4V	+2.0
Tantalum	+2.25
Titanium (pure)	+2.4
Ti-4.5Al-5Mo-1.5Cr	+2.4

*Breakdown varies and is in this range.

Anodic polarization curves for titanium and the three titanium alloys listed here are shown in figure 7 along with polarization curves for the other metals. Note the wide passive region. The effects of alloying elements are not great except in the case of the memory alloy which contains 50 percent nickel and the Ti-13Cu-4Ni alloy. The breakdown potential for pure nickel in Hanks' solution is less than +0.1 volt [16]. Even the nickel bearing titanium alloys were far more passive than the cobalt-chromium alloys or 316L stainless steel. Note that tantalum also has a high breakdown potential.

Repassivation Studies. The surface oxide film of a metal in service may be removed or disturbed by fretting, abrasion, cracking or scratching. The repassivation kinetics and the passive film stability become important if the material is to retain its corrosion resistance and integrity. Repassivation studies were conducted in Hanks' solution at 37 °C. The specimen, the Ti-6Al-4V alloy, was abraded with a SiC paper and had its passive film removed. The anodic current transient was recorded on an oscilloscope immediately following the passive film removal. Figure 8 shows the current transient at applied potentials of zero, +600 mV, +1200 mV and +1500 mV. All voltages in the repassivation studies are in reference to the saturated calomel electrode. Within this voltage range, there is little change in the repassivation rate as indicated by the peak height and decay. There also is little change in the film stability which is indicated by the leveling off of the current transient curve. At higher potentials, the (I/I_{max}) increases and the passive film becomes unstable. This is shown in figure 9 for potentials of +3.0 V, +4.0 V, +5.0 V, +6.0 V, and +7.0 V, respectively. Note that at +6.0 V, the steady state current is higher and the film becomes

unstable. The degree of passivation which occurs is expressed by $I\ /I_{max}$ is the steady state current and I_{max} is the maximum current as indicated in figure 9. When compared with the other metals of table 2, titanium alloys have, by far, the most stable surface film. For example films on 316L stainless steel become unstable at 0.2 V and those on Co-Cr-Mo become unstable at 0.42 V [18] while films on pure titanium are stable to 2.4 V.

CONCLUSIONS

The excellent corrosion resistance of titanium and titanium alloys in saline and physiological environments has been reaffirmed. Some specific conclusions relating to surface preparation and corrosion behavior of titanium are as follows:

1. Open circuit electrode potential measurements in Hanks' physiological solution indicate that titanium alloys will exhibit the same rest potential after a period of 2 to 3 weeks regardless of prior treatment. The surface film will change to approach an equilibrium state for the given solution.
2. Transmission electron micrographs show that the morphology of the titanium surface is affected by prior treatment. Washing in acid or alkaline solutions results in a roughened surface, an effect which is not observed at neutral pH.
3. Electron diffraction patterns along with the transmission electron micrographs show that the nature and composition of the titanium surface film change with variation of treatment. Alkaline washes produce a surface film consisting of a mixed oxide containing both titanium oxide and the alkali oxide.

Hot acid washes produce the surface oxide, TiO_2 anatase, and hot water or hot saline water washes result in a surface oxide which is TiO or a lower form of titanium oxide. Steam sterilization can produce TiO, lower oxides and occasional areas of TiO_2 anatase.

4. Anodic polarization measurements in Hanks' solution of numerous titanium alloys and other surgical implant metals show the titanium materials to be the most corrosion resistant with more positive breakdown potentials. This breakdown potential is dependent on alloy composition, with nickel acting to lower it.
5. Repassivation studies show that titanium and other surgical implant metals repassivate readily. The stability of the surface oxide film on titanium persists to +2.4 volts, which is +1.98 volts beyond the next most stable oxide on the Co-Cr-Mo alloy.

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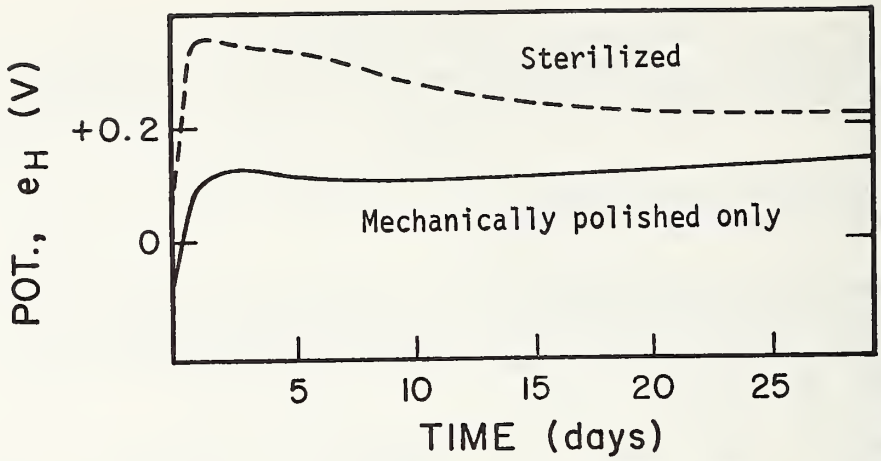

Fig. 1a. Open circuit potential vs. time curve for titanium, mechanically polished, steam sterilized and immersed in Hanks' solution.

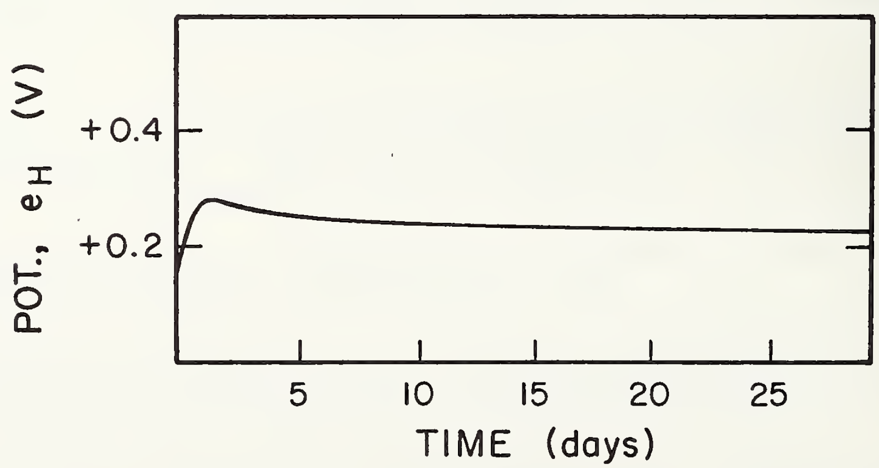

Fig. 1b. Open circuit potential vs. time curve for titanium, mechanically polished, washed in boiling 3.5% NaCl for 30 min., steam sterilized and immersed in Hanks' solution.

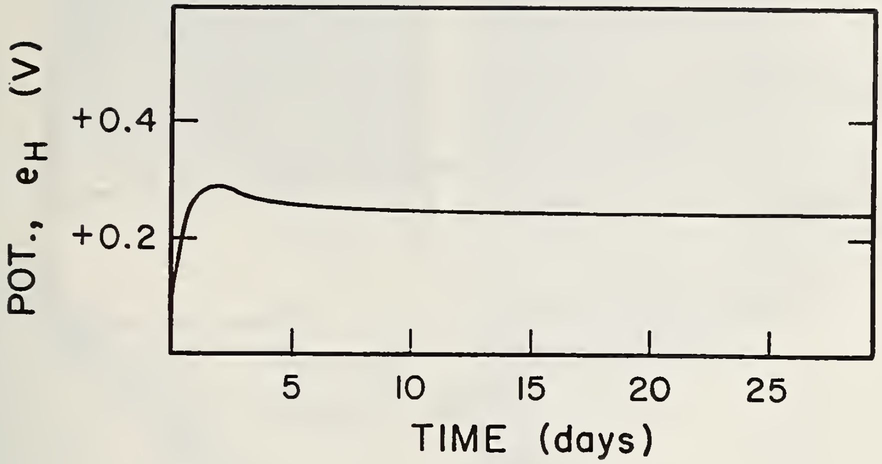

Fig. 1c. Open circuit potential vs. time titanium, mechanically polished, washed for 15 min. in 30% HNO_3 at 60°C, steam sterilized and immersed in Hanks' solution.

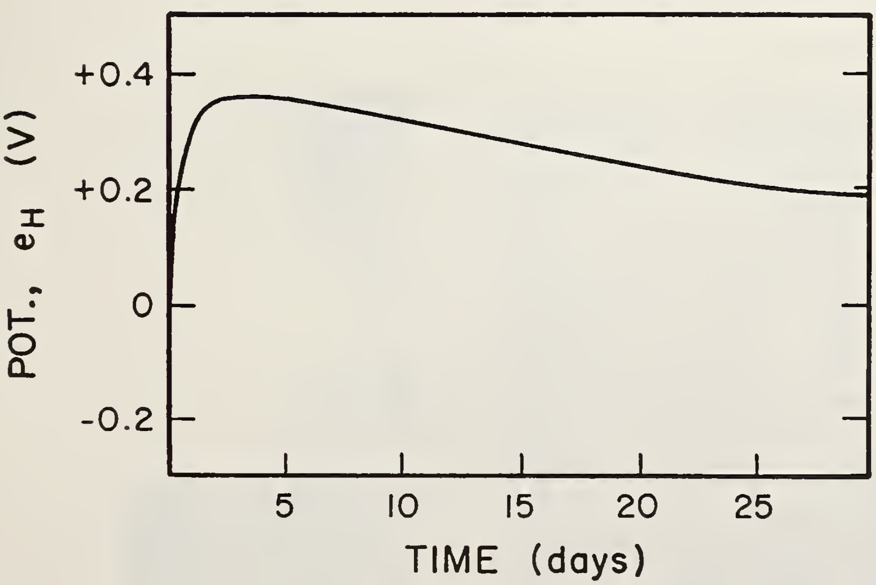

Fig. 1d. Open circuit potential vs. time curne for titanium, mechanically polished, washed for 2 min. in hot .5N NaOH, immersed in Hanks' solution.

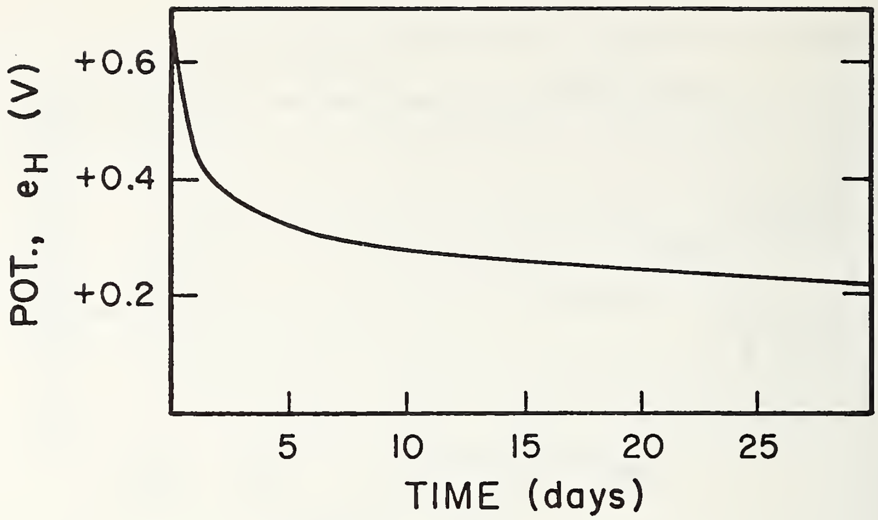

Fig. 1e. Open circuit potential vs. time curve for titanium, mechanically polished, washed 15 min. in 30% HNO_3 at 60°C, unsterilized and immersed in Hanks' solution.

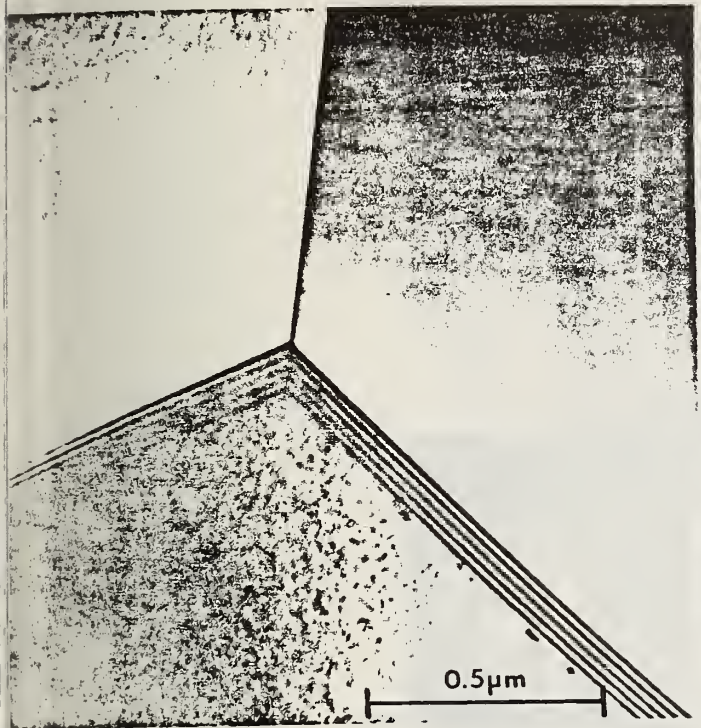

Fig. 2.

Titanium after electropolishing.

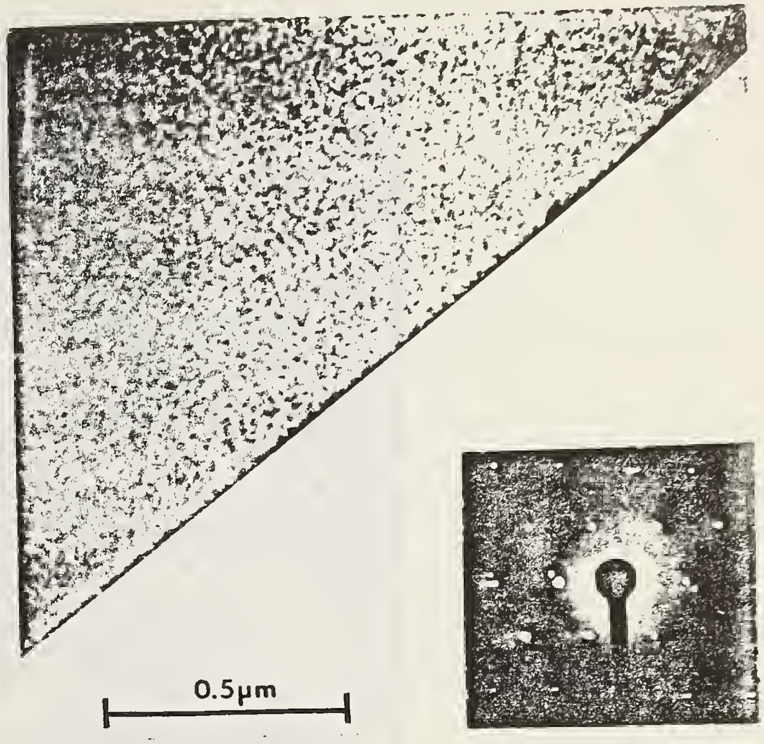

Fig. 3

Titanium after electropolishing, immersing in boiling water for 30 minutes and steam sterilizing. Diffuse ring indicates a surface film, probably a lower titanium oxide.

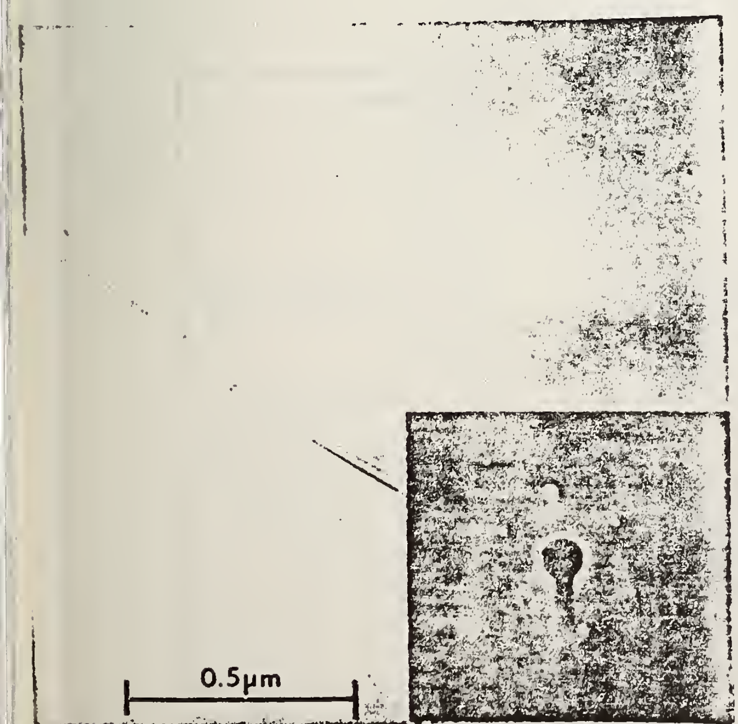

Fig. 4.

Titanium after immersing in boiling 3.5% NaCl for 30 min. and steam sterilizing. Diffuse rings indicate TiO.

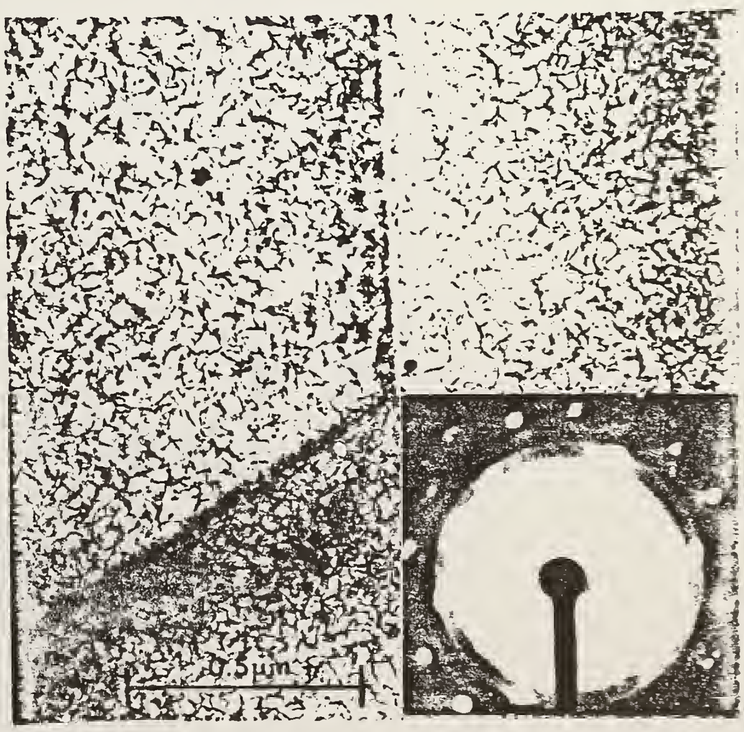

Fig. 5.

Titanium after immersing in hot .5N NaOH for 15 min. and steam sterilizing. Surface film is a form of $NaO{\cdot}XTiO_2$.

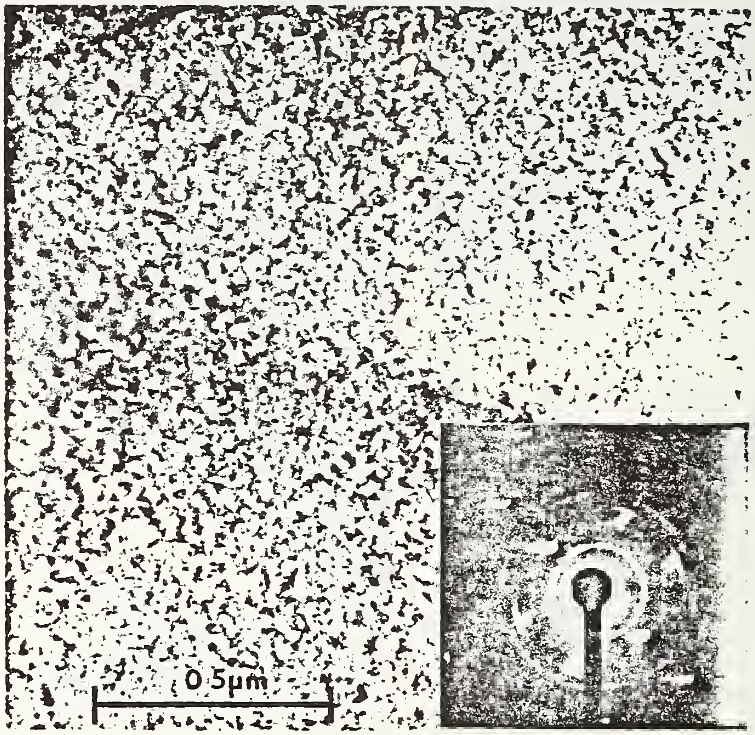

Fig. 6.

Titanium after immersing in hot 30% vol. HNO_3 for 30 min., and steam sterilizing. Surface film is TiO_2 anatase.

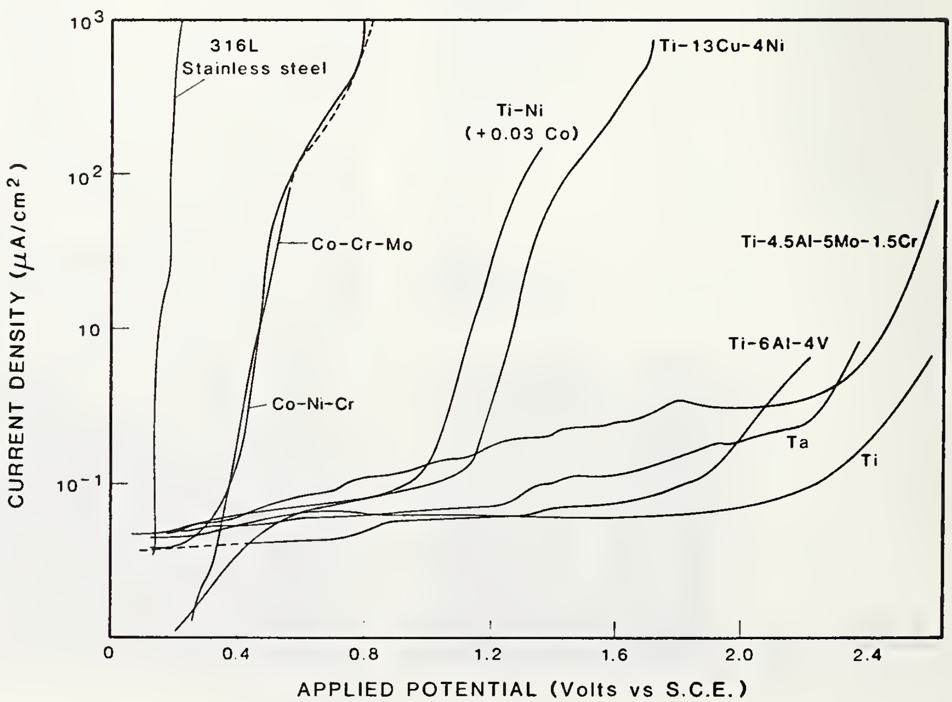

Fig. 7
Anodic polarization curves for titanium, titanium alloys and other implant metals. Potentiostatic measurements were made in Hanks' solution.

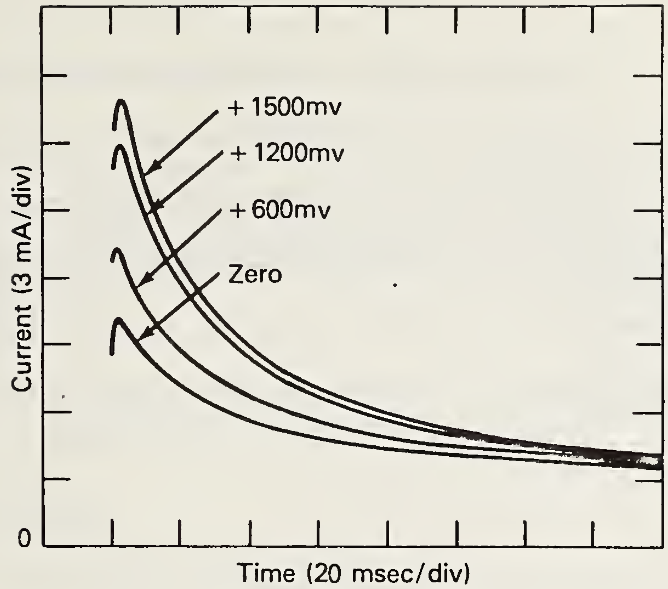

Fig. 8. Current (i) vs. Time (t) curves showing repassivation of Ti-6Al-4V at 0 (bottom curve), +600mV, +1200mV and +1500mV, respectively.

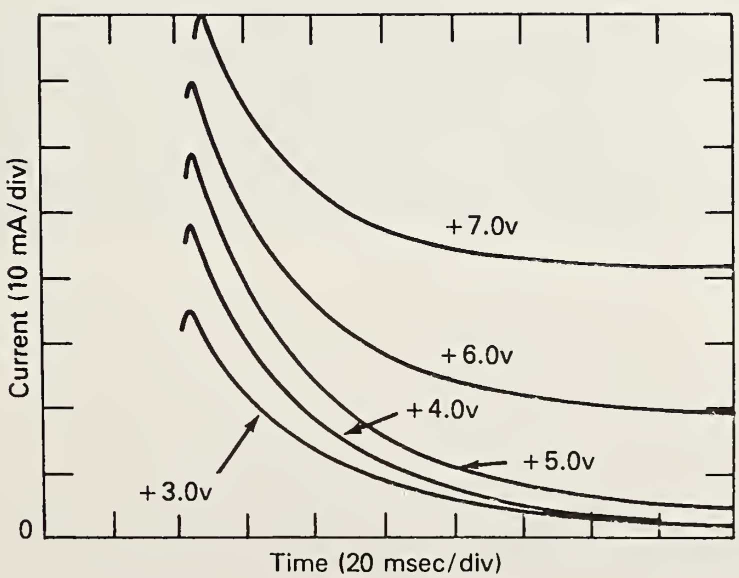

Fig. 9. Current (i) vs. Time (t) curves showing repassivation of Ti-6Al-4V at +3.0V (bottom curve), +4.0V, +5.0V, +6.0V and +7.0V, respectively.

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11. ABSTRACT (A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here)

A literature review covering articles on stress analyses in total hip replacement prostheses and the metal / bone cement interface is presented. The literature indicated the need for a test which utilized loading in torsion, and such a test was developed and is described. The test can be used to determine the influence of various parameters, including surface roughness and passivation and sterilization treatment on the strength of the metal/bone cement bond. Some preliminary tests were conducted and results are given. Future work using the mechanical test developed is discussed. Additional studies were conducted on the surface preparation of titanium, and data are presented to show that changes in initial electrochemical behavior and varying degrees of surface roughness occur depending on whether the metal receives a neutral, alkaline or acidic washing treatment.

12. KEY WORDS (Six to twelve entries; alphabetical order; capitalize only proper names; and separate key words by semicolons)

bone cement, hip prosthesis, stress analysis, surface preparation, surgical implant metals test method, titanium

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